

# THESIS

I. THE STRUCTURE OF  
OCTACHLOROCYCLOBUTANE.  $C_4Cl_8$

II. FURTHER STUDY OF THE  
STRUCTURE OF  $C_8F_{12}$

THOMAS BARRON OWEN

1950

Thesis  
095

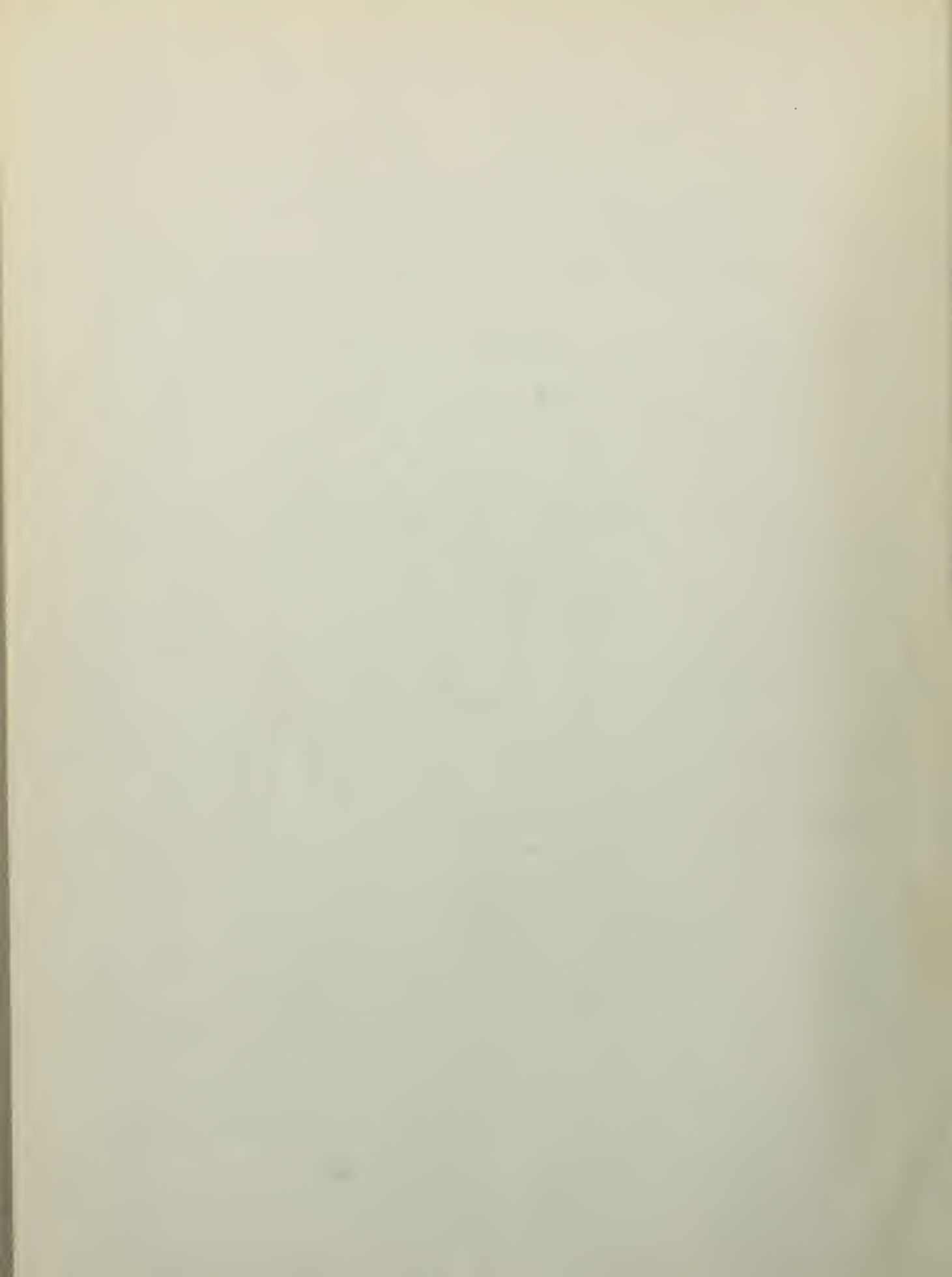
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- I THE STRUCTURE OF OCTACHLOROCYCLOBUTANE ,  $C_4Cl_8$   
II FURTHER STUDY OF THE STRUCTURE OF  $C_3F_{12}$

A Thesis

Presented to the Faculty of the Graduate School of  
Cornell University for the Degree of  
Doctor of Philosophy

By

Thomas Barron Owen

June, 1950

Thesis  
095

THE EFFECTS OF CRYSTALLINITY ON THE  
MECHANICAL PROPERTIES OF POLYETHYLENE

A Thesis  
Submitted to the Faculty of the Graduate School of  
Cornell University for the Degree of  
Doctor of Philosophy

By  
THOMAS BARTON DEAN  
June, 1960

## BIOGRAPHICAL SKETCH

The author was born on March 19, 1920 in Seattle, Washington. He attended the Seattle public schools and was graduated from Broadway High School in 1936. In June, 1940 he received the degree of Bachelor of Science in Chemical Engineering, cum laude. While employed by the Union Oil Company of California, he was called to active duty in the United States Navy in December of that year. After five years overseas service he returned to this country with the permanent rank of Lieutenant Commander, United States Navy. He studied electronic engineering for one year at the United States Naval Postgraduate School, Annapolis, Maryland and then entered Cornell University in 1947.

He was married to Rosemary Stolz in December, 1944 and has two children, Catherine Adams and Thomas Jr. of ages four and two respectively.





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In future assignments it is felt that the experience he has gained here will serve to increase the mutual respect and understanding of the scientist and the naval officer.

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Cornell University.

In future assignments he is sure that the opportunity  
will be given him to serve in various capacities  
and understanding of the subject and the  
social officer.



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PART I

THE STRUCTURE OF OCTACHLOROCYCLOBUTANE ,  $C_4Cl_8$

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## INTRODUCTION

During the past few years Professor W. T. Miller and his students have been engaged in the preparation and study of a variety of fluoro- and chlorocarbons. One of the most interesting of these is the saturated dimer of hexafluorobutadiene,  $C_8 F_{12}$ ,<sup>(10)</sup> the structure of which is to be discussed in part II of this thesis. X-ray studies of this compound thus far have been predicated on a molecular model possessing a center of symmetry which, if it were to exist, would greatly simplify the structure problem. Since  $C_8 F_{12}$  is thought to contain three fused cyclobutane rings it would be desirable to know whether or not a simple cyclobutane structure has a plane or puckered ring. If puckering of the ring were confirmed, it would be reasonable to assume that the molecule of  $C_8 F_{12}$  has no center of symmetry.

In reviewing the literature it is found that spectroscopic studies of cyclobutane and of its fluorinated analogue  $C_4 F_8$  have been interpreted as indicating the carbon ring to be planar. Dunitz<sup>(8)</sup> in the X-ray study of one form of tetraphenyl cyclobutane found the structure to have a planar ring also. He states however that an isomer of this compound may have a fourfold alternating axis of symmetry ( $S_4$ ). However Livingston<sup>(14)</sup>





in a private communication to this Department has stated that preliminary evidence from the electron diffraction investigation of  $C_4F_8$  indicates a puckered ring with unusually long carbon - carbon distances.

Recently Mr. Fred McLafferty working under Professor Miller's direction has prepared octochlorocyclobutane,  $C_4Cl_8$ , thus making it available for X-ray studies. This presented an excellent opportunity to establish the configuration of the four-membered carbon ring so as to obtain ideas of the probable structure of  $C_8F_{12}$ .

Part I of this dissertation concerns itself with the investigation of the structure of  $C_4Cl_8$ .

in a private communication to this Department has stated that preliminary evidence from the electron microscope investigation of CP 74 indicates a structure that may be reversibly torn rather than random disorder.

Recently Dr. Paul Callaghan, working under Professor Miller's direction has prepared approximately 1000 CP 74 films making it possible to study the structure. This presented an excellent opportunity to establish the configuration of the two-dimensional sheets and as an indication of the general structure of CP 74.

Part I of this discussion concerns itself with the investigation of the structure of CP 74.

The first part of the discussion deals with the structure of CP 74 and is divided into two sections. The first section deals with the structure of CP 74 and the second section deals with the structure of CP 74.

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The second part of the tenth section deals with the structure of CP 74 and is divided into two parts. The first part deals with the structure of CP 74 and the second part deals with the structure of CP 74.

## EXPERIMENTAL PROCEDURE

### Preparation and Mounting of Samples.

As remarked above, the compound was prepared by Mr. Fred McLafferty. Essentially the synthesis consists of chlorinating butadiene to give a chlorobutene which in the presence of  $AlCl_3$  at  $100^\circ C.$  gives several products, one of which is  $C_4Cl_3$ . The material is separated and purified by recrystallization from methyl alcohol.

A portion of the sample was dissolved in benzene in a small test tube and allowed to crystallize slowly overnight. Clear flat needle-like crystals were obtained which when examined microscopically between crossed Nicol prisms showed continuous extinction parallel to the needle axis irrespective of any angular rotation about this axis. Nevertheless, it later appeared that the needle axis was not the unique symmetry axis of the monoclinic crystal.

A needle approximately 0.3 mm. in diameter was mounted vertically on the goniometer head such that the needle axis coincided with the rotation axis. Since the compound has an appreciable vapor pressure it was necessary to dip the crystal in a solution of Canada balsam in xylene to form a protective coating which

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A portion of the sample was dissolved in benzene  
in a small test tube and allowed to crystallize slowly  
overnight. Clear flat needle-like crystals were obtained  
which when examined microscopically between crossed  
polarizers showed continuous extinction parallel to  
the needle axis irrespective of any regular rotation  
about this axis. Invertin, if later specimens that  
the crystals are not the same symmetry axis of the  
molecular repeat.

A needle approximately 0.1 mm. in diameter was  
mounted vertically on the polarimeter head with fast  
the needle axis coincided with the rotation axis. Since  
the compound was an optically active substance it was  
necessary to tilt the crystal in a position of constant  
rotation in order to have a positive rotation which



would prevent sublimation. Prior to photography coarse adjustments for alignment were made on a one circle goniometer.

#### Photographic Recording of Diffraction Data.

Oscillation photographs were taken and the method of Hendershot<sup>(12)</sup> was employed to align the crystal for use on the Weissenberg instrument. The oscillation pictures indicated a spacing of  $0.3 \text{ \AA}$  along the needle axis which was tentatively designated as the  $c$  axis.

The zero and four higher layer equi-inclination Weissenberg photographs of this crystal were obtained using  $\text{Cu K}\alpha$  radiation. The observed symmetry of the X-ray diffraction effects was  $C_{2h} - 2/m$  with the monoclinic axis lying in a plane normal to the needle axis. Only  $(0k0)$  reflections for  $k$  odd were missing. The following space groups satisfy these conditions:  $C_2^2 - P2_1$  or  $C_{2h}^2 - P2_1/m$ . The latter has a center of symmetry, whereas the former does not.

To obtain sufficient data for structure analysis it is necessary to observe reflections for rotation about at least one other axis. Since the unit cell is monoclinic, the unique axis which lies in the cross section of the needle-like crystals could be located. Sections perpendicular to the needle axis were cut out

would be very similar. Since the photographs were  
 adjusted for alignment with the axis  
 of symmetry.

The photograph showing the distribution of the  
 oscillation photographs were taken and the  
 of (symmetry) was compared to the axis for  
 use as the reference instrument. The oscillation  
 picture indicated a spacing of  $\sim 1.5 \times$  along the axis  
 axis which was tentatively designated as the  $z$  axis.  
 The axis was then slightly tilted-inclination  
 relationship photographs of this crystal were obtained  
 using the method. The observed symmetry of the  
 X-ray diffraction effect was  $C_{2h} = 2/m$  with the same  
 clinic axis lying in a plane normal to the needle axis.  
 Only (hkl) reflections for  $h$  odd were missing. The  
 following space groups satisfy these conditions:  
 $C_{2h} = C_{2h} = C_{2h} = C_{2h}$ . The latter has a center of  
 symmetry, whereas the former does not.  
 It seems sufficient that the structure analysis  
 is necessary to compare reflections for rotation  
 about at least one crystal axis. Since the unit cell  
 is monoclinic, the unique axis which lies in the cross  
 section of the needle-like crystals could be located.  
 sections perpendicular to the needle axis were cut out

using a sharp razor blade. The approximate orientation of the monoclinic axis was known from a comparison of the symmetry of the reflections obtained on the original oscillation photographs with the crystal position. Again oscillation pictures were used for alignment and the zero and six upper layer equi-inclination Weissenberg photographs were obtained for rotation about the monoclinic axis, b. The triple film technique was used throughout.

#### Lattice Constants.

The lattice constants were determined from the two zero layer Weissenberg photographs (i.e. around b and c) upon which reflections from NaCl had been superimposed. The monoclinic angle was determined by the method of angular lag as described by Buerger<sup>(5)</sup> and by direct measurement on the zero layer b-axis film. Final values are:

$$\underline{a} = 8.00 \text{ \AA.}$$

$$\underline{b} = 10.64$$

$$\underline{c} = 6.28$$

$$\beta = 107^{\circ}45'$$

#### Molecules per Unit Cell.

Although no pycnometric density has been measured for this compound, it was reasonable to assume it roughly







as 2 gm./cc. The volume of the unit cell is  $509.5 \text{ \AA}^3$ .

Thus from the formula

$$= 1.660 \text{ nM/V}$$

where  $n$  = number of molecules per unit cell

$M$  = molecular weight

$V$  = volume of unit cell in  $\text{\AA}^3$

we get, for  $n=2$ , an X-ray density of 2.16 gm./cc.

#### Measurement of Intensities.

The reflections were indexed and the value of  $\sin \theta$  for each spot was determined graphically from construction of the reciprocal lattice. The method is described by Bunn.<sup>(6)</sup> Relative intensities for each film of each layer photograph were estimated by visual comparison with a calibrated intensity scale having spots of a size comparable to those appearing on the films and with the following range of intensities:

2:3:5:6:9:11:13:15:19:22:26:30:35:40:42:43:50:57:64:76:  
79:82:88:100:118:130:139:145:147.

The value of 2 indicated a spot that was just visible whereas that of 147 indicated near saturation. The use of the triple film technique permitted a good estimation of intensities over a wide range. The values obtained were corrected for the Lorentz and Polarization factor and the Tunell<sup>(19)</sup> factor where applicable to obtain relative  $|F_{hkl}|^2$  values.

at 2 per cent. The volume of the unit cell is 0.00125  
Take from the formula  
$$V = 1,400 \text{ m}^3$$
  
where n = number of molecules per unit cell  
M = molecular weight  
$$V = \text{volume of unit cell in } \text{\AA}^3$$
  
we get, for unit cell density of 2.14 g./cc.  
Measurement of intensities.

The reflections were indexed and the value of sin  $\theta$   
for each spot was determined graphically from a series  
film of the specimen studied. The method is described  
by Moore. (6) Relative intensities for each film of each  
layer photograph were obtained by visual comparison with  
a calibration intensity scale having spots of a size com-  
parable to those appearing on the film and with the  
following range of intensities:  
50:40:30:20:10:5:2.5:1.25:0.625:0.3125:0.15625:0.078125  
75:62.5:50:37.5:25:12.5:6.25:3.125:1.5625:0.78125:0.390625

The value of  $\lambda$  indicated a spot that was just visible  
whereas that of 50 indicated near saturation. The way  
of the film between positions a good reflection  
of intensities over a wide range. The values obtained  
were corrected for the Lorentz and polarization factors  
and the result (19) factor which applies to obtain  
relative values.

The zero layer Weissenberg photograph about  $c$  was taken as the standard film and by careful cross-calibration all relative  $|F_{hk\ell}|^2$  were reduced to the common level. The factors by which the observations from the respective films were multiplied to reach the common level are listed below:

$c_1$	1.18	$b_0$	4.15
$c_2$	1.23	$b_1$	1.52
$c_3$	1.82	$b_2$	1.54
$c_4$	2.86	$b_3$	1.25
		$b_4$	0.95
		$b_5$	1.43
		$b_6$	1.43

where the letter denotes the axis of rotation and the subscript the layer line.

The effects of absorption were small enough to be neglected.





## DETERMINATION OF ATOMIC POSITIONS

All summations described here were calculated using Beavers and Lipson<sup>(2)</sup> strips.

Patterson Projection,  $P(X,Y)$ .

Since  $hk0$  data were first available the Patterson projection,  $P(X,Y)$  on the  $(001)$  plane was made.

For the diffraction symmetry for this crystal

$$|F_{hk0}|^2 = |F_{\bar{h}k0}|^2 = |F_{h\bar{k}0}|^2 = |F_{\bar{h}\bar{k}0}|^2;$$

and it can be shown that

$$\begin{aligned} P(X,Y) = & 4 \sum_{h=1}^{\infty} \sum_{k=1}^{\infty} |F_{hk0}|^2 \cos 2\pi hX \cos 2\pi kY \\ & + 2 \sum_{h=1}^{\infty} |F_{h00}|^2 \cos 2\pi hX \\ & + 2 \sum_{k=1}^{\infty} |F_{0k0}|^2 \cos 2\pi kY + |F_{000}|^2. \end{aligned}$$

This projection was used to determine with which of the two space groups,  $P2_1$  or  $P2_1/m$ , the data were consistent. If the former is correct,  $P(X,Y)$  should have peaks at  $(2x, 1/2)$  for each pair of equivalent atoms in the cell as well as peaks due to vectors between non-equivalent atoms.

If the space group  $P2_1/m$  is correct, atoms lying in the planes of symmetry occupy positions:

(e)  $x, 1/4, z; \bar{x} 3/4, \bar{z}$ . For atoms in fourfold positions there are the following positions:

All elements defined here were obtained using  
 Gauss and Legendre's method.

Let  $\mathcal{H}$  be the Hilbert space.

Then one can find a unique element  $\mathcal{H}$  such that  
 the projection  $\mathcal{H}$  on  $\mathcal{H}$  is zero.

For the orthogonal system for this space

$$\| \mathcal{H} \| = \| \mathcal{H} \| = \| \mathcal{H} \| = \| \mathcal{H} \|$$

and it can be shown that

$$\| \mathcal{H} \| = \| \mathcal{H} \| = \| \mathcal{H} \| = \| \mathcal{H} \|$$

$$\| \mathcal{H} \| = \| \mathcal{H} \| = \| \mathcal{H} \| = \| \mathcal{H} \|$$

$$\| \mathcal{H} \| = \| \mathcal{H} \| = \| \mathcal{H} \| = \| \mathcal{H} \|$$

This projection was used to determine the value

of the space group  $\mathcal{H}$  at  $\mathcal{H}$ , and the value

obtained. It is known that  $\mathcal{H}$  is zero.

have been at  $\mathcal{H}$  for some time at various points

in the cell as well as points in the space group.

equivalent points.

If the space group  $\mathcal{H}$  is known, then the

in the plane of symmetry group  $\mathcal{H}$ .

Let  $\mathcal{H}$  be the space group. The space is known to be

the following conditions:

(f)  $x, y, z; \bar{x}, \bar{y}, \bar{z}; x, 1/2 - y, z; \bar{x}, y + 1/2, \bar{z}$ . Vectors between equivalent atoms occupying twofold positions will give rise to peaks at  $(2x, 1/2)$  whereas those between equivalent atoms occupying fourfold positions will give rise to peaks at:

$$\begin{aligned} &2\bar{x}, 2\bar{y}; 0, 1/2 - 2y; 2\bar{x}, 1/2; 2x, 1/2; 0, 2y + 1/2; 2\bar{x}, 2y; \\ &2x, 2y; 0, 2y - 1/2; 2x, -1/2; 2\bar{x}, -1/2; 0, -2y - 1/2; 2x, 2\bar{y}. \end{aligned}$$

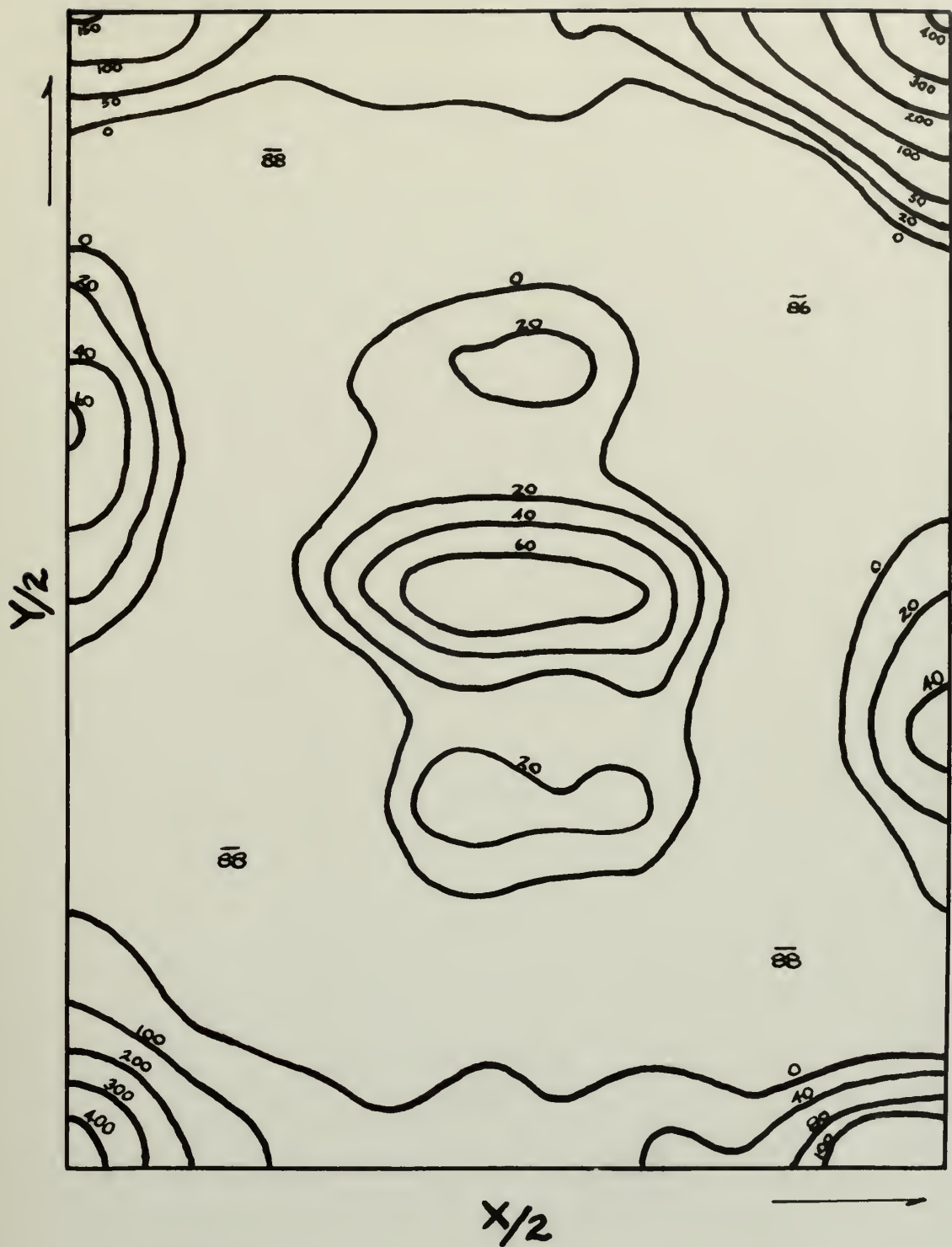
The projection is shown in Figure 1. Only one-fourth the plane is shown since the other quarters are related to the first by two mutually perpendicular mirror planes as can be seen from a consideration of the Patterson function.

If the centrosymmetrical group be correct and if one considers the orientation of a model of the molecule with respect to the symmetry plane, he should be able to predict the positions of compatible peaks in the Patterson. If the ring lay in the plane one would expect a strong Cl-Cl peak at  $X=0, Y=0.3$  but such an orientation would give no peak at  $X=0, Y=0.5$ . If no atoms lay in the symmetry plane the molecule would have to be oriented such that the symmetry plane bisected the parallel legs of the carbon ring. Again one would expect a strong Cl-Cl peak along  $X=0$  but not at  $X=0, Y=1/2$ . The last possibility is that the diagonal of the carbon ring could lie in the symmetry plane.









PATTERSON PROJECTION

$P(x, y)$

Figure 1



If a scaled molecular model is so oriented and allowed to pucker it can be seen that the last configuration would give rise to two different Cl-Cl separations parallel to the unique axis. The shorter Cl-Cl vector would cause a peak at  $X=0$  somewhere between  $Y=1/4$  and  $Y=1/2$ . The longer vector would show up at  $X=0$  and near  $Y=1/2$ . Such maxima do appear in the projection so this orientation of the molecule is consistent thus far. The peak at  $X=1/2$ ,  $Y=0.19$  if caused by a set of equivalent fourfold atoms would also mean a peak at  $X=0$   $y=0.31$  (i.e.  $2x=1/2$ ,  $2y=0.19$  corresponds to  $0, 0.50-0.19$  or  $0, 0.31$ ) if one considers the list above. Also, if the peak at  $X=1/2$ ,  $Y=1/2$  included a vector due to the other set of fourfold chlorines one would expect a peak at  $X=0$ ,  $Y=1/2$  which indeed is obtained. The chosen position of the molecule thus fits in with the interpretation of the Patterson and all peaks can be explained. The projection is then consistent with the space group,  $P2_1/m$ .

It was possible to assign  $x$  and  $y$  parameters to the chlorine atoms of the molecule and by assuming normal C-C bond lengths to assign parameters to the carbons as well. These were:

$x$	$y$	$x$	$y$	$x$	$y$
$C_1$ +0.125	$1/4$	$Cl_1$ +0.235	+0.092	$Cl_6$ +0.045	$1/4$
$C_2$ +0.250	+0.156	$Cl_4$ +0.255	+0.255	$Cl_7$ -0.075	$1/4$
$C_3$ +0.375	$1/4$	$Cl_5$ +0.465	$1/4$	$Cl_8$ +0.565	$1/4$

NOTA: Este documento es el trabajo voluntario de la UCLM

allowed to pocket it can be seen that the last assignment  
tion would give rise to the different  $CI$  assignments  
parallel to the edge sets. The classes  $CI$  would  
could cause a peak at the maximum between  $Y_{1/2}$  and  
 $Y_{1/2}$ . The longer edge sets would show up at  $Y_{1/2}$  and not  
 $Y_{1/2}$ . When making an appeal to the projection to this  
extension of the analysis is necessary to see this. The  
case at  $Y_{1/2}$ ,  $Y_{1/2}$  is known by a set of conditions

IT-Dev G&S Pa Acct & mkt vald klipp avrds klippad

(11.9.0 to 14.0=25.0, 0 to 25.0=25.0, 25.0 to 50.0=50.0, 50.0 to 75.0=75.0, 75.0 to 100.0=100.0)

It was because the first show, also, it has been at

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Could anyone see a way to make this work?

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08760900 and all books can be explained. The evidence

It then consisted with the same group.

It was possible to obtain a red concentrate in the

collaborative aspect of this initiative and the resulting impact.

→ Local limits to avoid overstate to the nation

THE UNIVERSITY OF CHICAGO

$\gamma$	$\theta$	$\gamma$	$\theta$	$\gamma$	$\theta$
$\sqrt{2}$	$0.0 \pm 0.00$	$0.0 \pm 0.00$	$0.0 \pm 0.00$	$1.0$	$0.125$
$\sqrt{2}$	$0.0 \pm 0.00$	$0.0 \pm 0.00$	$0.0 \pm 0.00$	$0.100$	$0.100$
$\sqrt{2}$	$0.0 \pm 0.00$	$1.0$	$0.0 \pm 0.00$	$1.0$	$0.175$



It might be well to describe the configuration of the molecule as postulated. In the symmetry plane the four chlorines form an isosceles trapezoid. The perpendicular bisector of the bases of this quadrilateral lies almost along the trace of the plane  $x=1/4$  in the symmetry plane. The four chlorine atoms out of the plane also form a trapezoid the plane of which is almost coincident with  $x=1/4$ . Figure 2 is a sketch of the molecule and will serve to clarify the above. The puckering of the ring can be seen.

#### Amplitude Calculations.

For the space group  $P2_1/m$  it can be shown that the contribution to the reflection amplitude  $F_{hk\ell}$  from two like atoms occupying twofold positions is

$$\begin{aligned} A &= 2fa \cos 2\pi(hx + \ell z) \cos \frac{\pi k}{2} \text{ for } k \text{ even} \\ &= -2fa \sin 2\pi(hx + \ell z) \sin \frac{\pi k}{2} \text{ for } k \text{ odd} \end{aligned}$$

where  $fa$  is the atomic scattering power of the atom. The contribution from four like atoms occupying fourfold positions is

$$\begin{aligned} A &= 4fa \cos 2\pi(hx + \ell z) \cos 2\pi ky \text{ for } k \text{ even} \\ \text{and } A &= -4fa \sin 2\pi(hx + \ell z) \sin 2\pi ky \text{ for } k \text{ odd.} \end{aligned}$$

From these relations the following relations can be deduced:

$$F_{hk\ell} = F_{\overline{h}\overline{k}\overline{\ell}} \text{ for all } k$$

$$F_{hk\ell} = F_{h\overline{k}\ell} \text{ for } k \text{ even}$$

$$F_{hk\ell} = -F_{h\overline{k}\ell} \text{ for } k \text{ odd.}$$

It might be well to describe the distribution of the molecules as follows. In the vicinity of the four chlorine atoms an electron is present. The positive charges of the atoms of this quadrilateral are almost along the lines of the plane and the plane also. The four chlorine atoms are at the plane also. From a perspective the plane of atoms is almost rectangular with sides. Figure 2 is a sketch of the molecule and will serve to clarify the above. The numbering of the atoms may be seen.

#### Empirical Calculations.

For the space group  $C_{2v}$  it can be shown that the contribution to the reflection amplitudes  $F(h, k, l)$  from the four atoms occupying positions is

$$F(h, k, l) = 4f \cos \frac{1}{2} \pi (h + k + l) \cos \frac{1}{2} \pi (h - k) \cos \frac{1}{2} \pi (h + k - l) \\ = 4f \cos \frac{1}{2} \pi (h + k + l) \cos \frac{1}{2} \pi (h - k) \cos \frac{1}{2} \pi (h + k - l)$$

where  $f$  is the atomic scattering power of the atom. The contribution from the four atoms occupying positions is

$$F(h, k, l) = 4f \cos \frac{1}{2} \pi (h + k + l) \cos \frac{1}{2} \pi (h - k) \cos \frac{1}{2} \pi (h + k - l) \\ = 4f \cos \frac{1}{2} \pi (h + k + l) \cos \frac{1}{2} \pi (h - k) \cos \frac{1}{2} \pi (h + k - l)$$

From these relations the following relations can be obtained:

$$F(h, k, l) = 4f \cos \frac{1}{2} \pi (h + k + l) \cos \frac{1}{2} \pi (h - k) \cos \frac{1}{2} \pi (h + k - l) \\ F(h, k, l) = 4f \cos \frac{1}{2} \pi (h + k + l) \cos \frac{1}{2} \pi (h - k) \cos \frac{1}{2} \pi (h + k - l) \\ F(h, k, l) = 4f \cos \frac{1}{2} \pi (h + k + l) \cos \frac{1}{2} \pi (h - k) \cos \frac{1}{2} \pi (h + k - l)$$

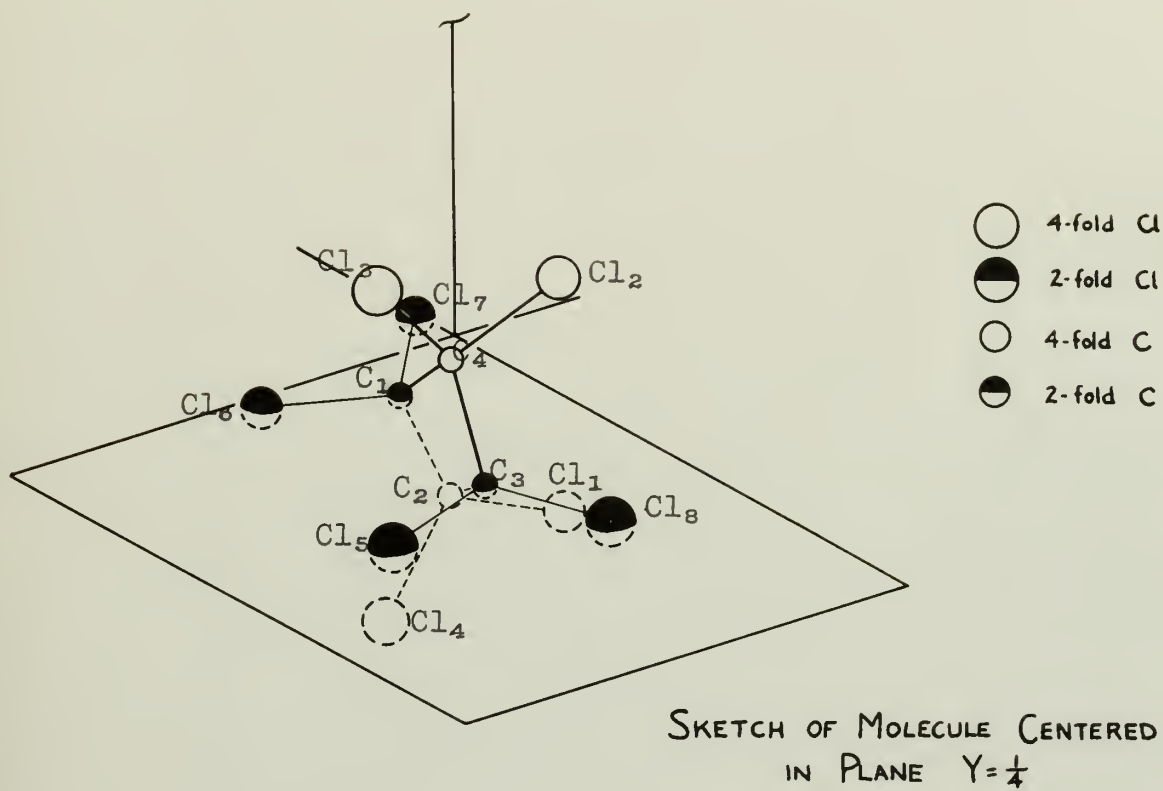
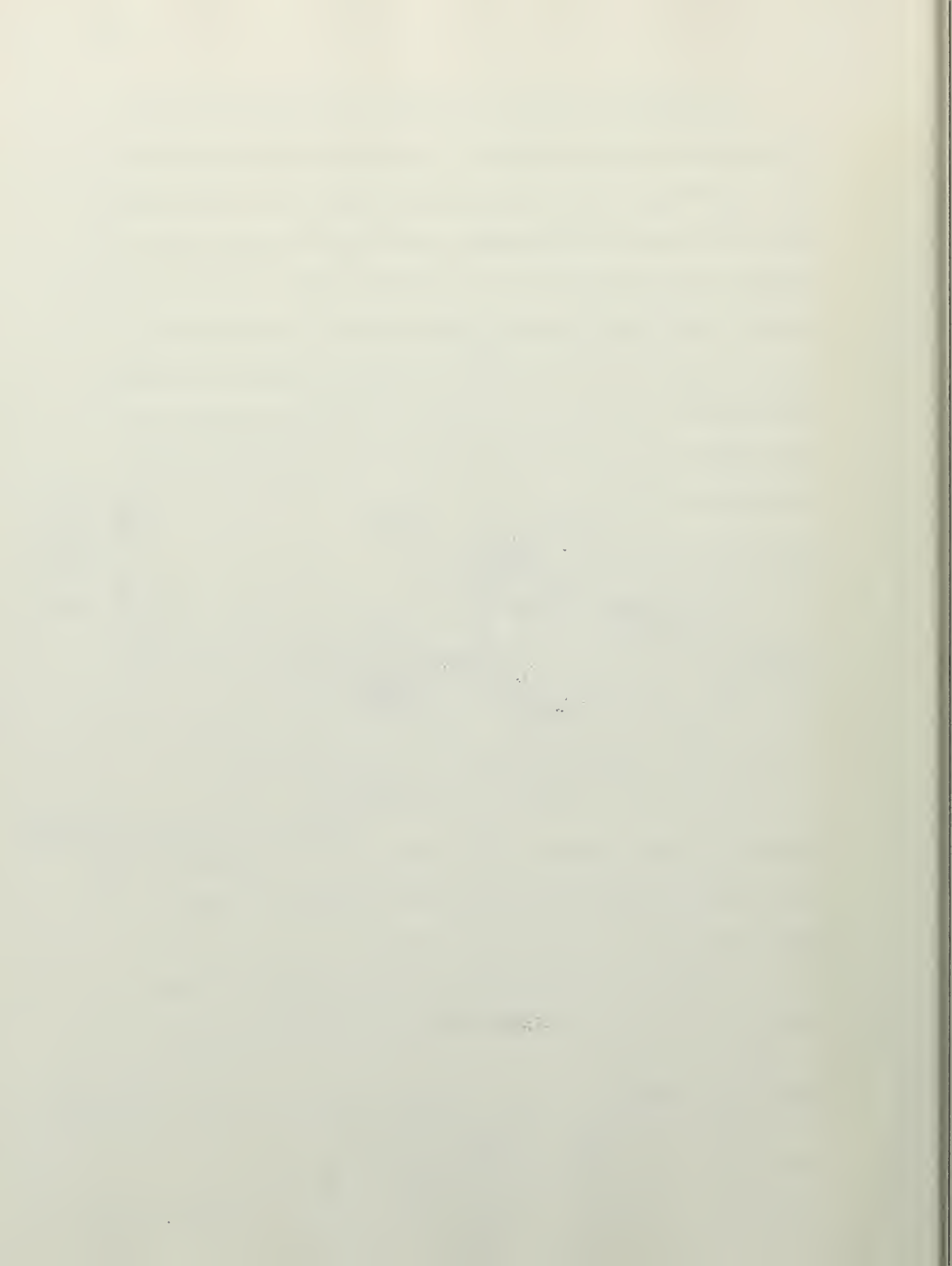


Figure 2





Fourier Projection,  $\rho(x,y)$ .

Using the trial parameters obtained from the interpretation of the Patterson, the phases for (hk0) data were calculated in order to make the Fourier projection,  $\rho(x,y)$ , on (001). It can be shown that

$$\begin{aligned} K \rho(x,y) = & 4 \sum_{h=1}^{\infty} \sum_{\substack{k=2n \\ n \geq 1}}^{\infty} F_{hk0} \cos 2\pi hx \cos 2\pi ky \\ & + 2 \sum_{h=1}^{\infty} F_{h00} \cos 2\pi hx + 2 \sum_{\substack{k=2n \\ n \geq 1}}^{\infty} F_{0k0} \cos 2\pi ky \\ & - 4 \sum_{h=1}^{\infty} \sum_{\substack{k=2n+1 \\ n \geq 0}}^{\infty} F_{hk0} \sin 2\pi hx \sin 2\pi ky \end{aligned}$$

(neglecting constant additive terms).

The projection is shown in figure 3. The positions of maxima were not too well resolved, but sufficiently so to confirm the postulated model. Chlorine peaks superimpose to some extent but are clear enough to obtain parameters. Carbon peaks are lost in those of the chlorines so carbon positions must be assigned from consideration of known C-C and C-Cl bond lengths. Atomic coordinates now became:

x	y	x	y	x	y
C <sub>1</sub> +0.100	1/4	Cl <sub>1</sub> +0.238	+0.095	Cl <sub>6</sub> +0.038	1/4
C <sub>2</sub> +0.234	+0.159	Cl <sub>4</sub> +0.245	+0.020	Cl <sub>7</sub> -0.080	1/4
C <sub>3</sub> +0.383	1/4	Cl <sub>5</sub> +0.470	1/4	Cl <sub>8</sub> +0.568	1/4

Fourier projection,  $\rho(x, y)$ .

Using the trial parameters obtained from the inter-

pretation of the Patterson, the phases for (hkl) data were calculated in order to make the Fourier projection.

$\rho(x, y)$  in (GDI). It can be shown that

$$\rho(x, y) = \frac{1}{V} \sum_{h=1}^N \sum_{k=1}^N \sum_{l=1}^N F(h, k, l) \exp(i\pi h x) \exp(i\pi k y) \exp(i\pi l z)$$

$$+ \frac{1}{V} \sum_{h=1}^N \sum_{k=1}^N \sum_{l=1}^N F(h, k, l) \exp(i\pi h x) \exp(i\pi k y) \exp(i\pi l z)$$

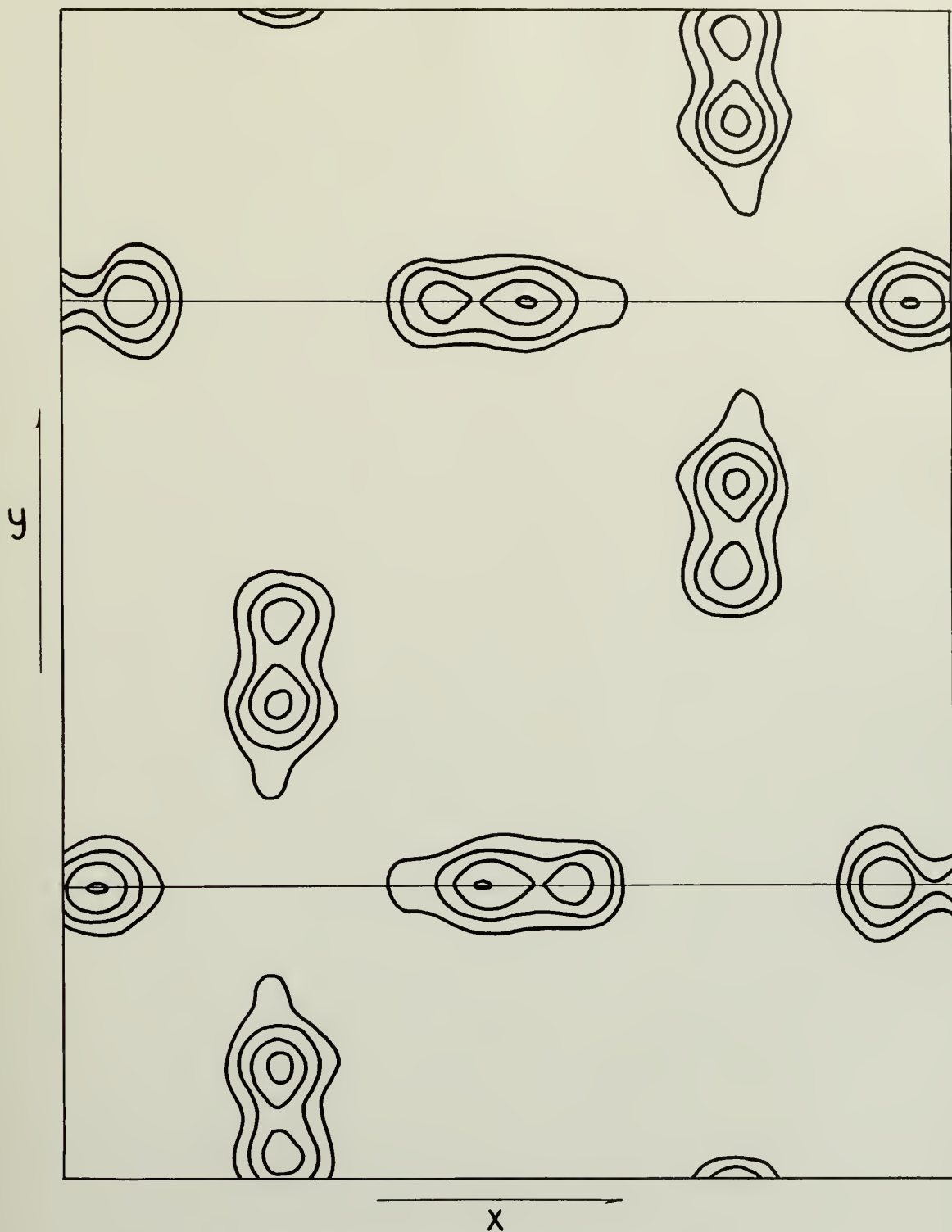
$$- \frac{1}{V} \sum_{h=1}^N \sum_{k=1}^N \sum_{l=1}^N F(h, k, l) \exp(i\pi h x) \exp(i\pi k y) \exp(i\pi l z)$$

(neglecting constant additive terms).

The projection is shown in Figure 2. The positions of maxima were not too well resolved, but sufficiently so to confirm the postulated model. Chlorine peaks appear to some extent but are clear enough to obtain parameters. Carbon peaks are lost in those of the chlorines as carbon positions must be assigned from consideration of known C-C and C-Cl bond lengths. Atomic coordinates

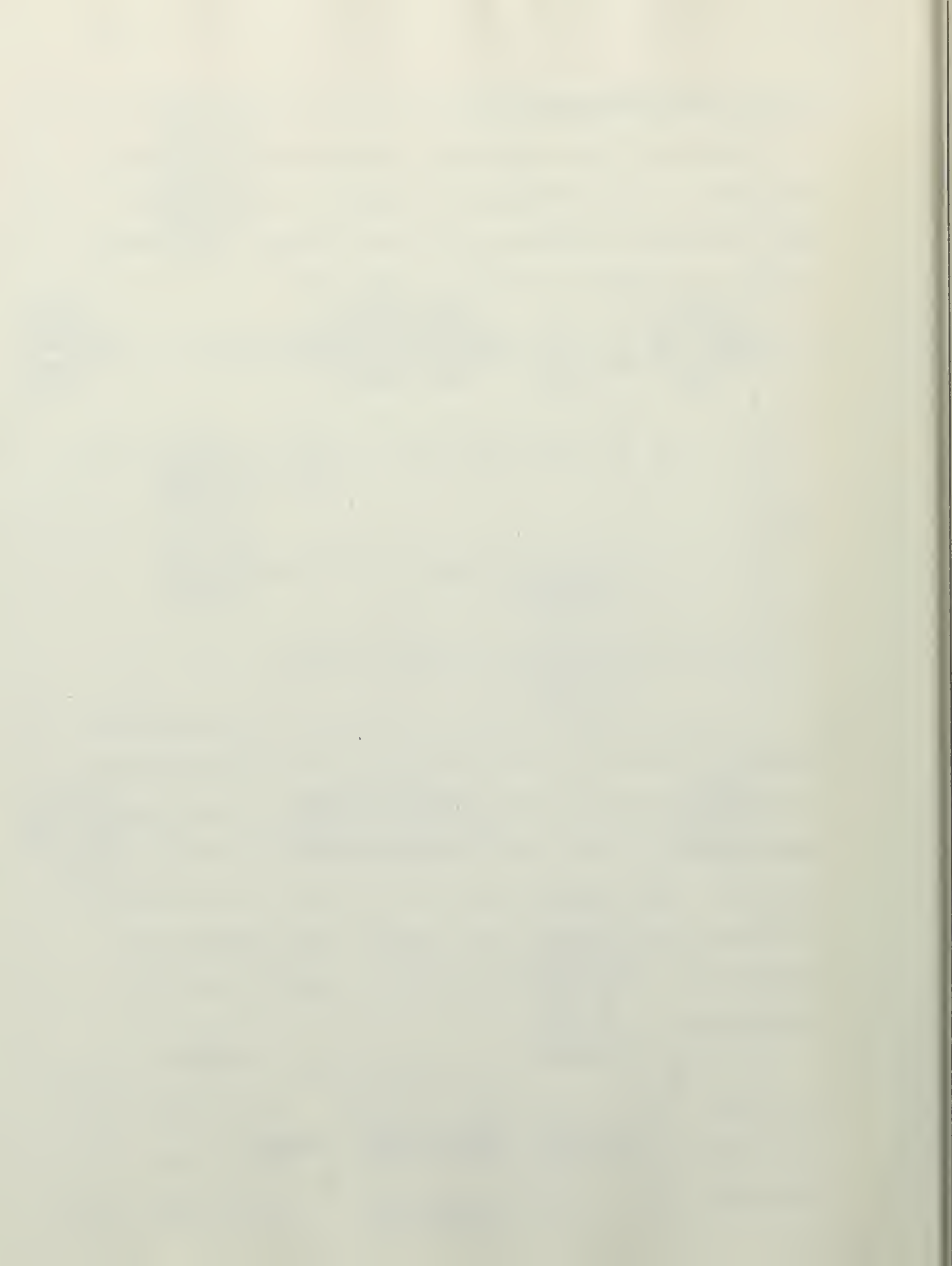
now become:

x	y	x	y	x	y
Cl <sup>1</sup> +0.100	1/4	Cl <sup>1</sup> +0.258	+0.092	Cl <sup>2</sup> +0.038	1/4
Cl <sup>2</sup> +0.234	+0.136	Cl <sup>3</sup> +0.262	+0.020	Cl <sup>4</sup> +0.080	1/4
Cl <sup>3</sup> +0.282	1/4	Cl <sup>4</sup> +0.470	1/4	Cl <sup>5</sup> +0.268	1/4



FOURIER PROJECTION  $p(x,y)$

Figure 3





### Orientation Along z.

At this stage, x and y coordinates were fairly well determined. The next problem was to determine positions along z. In effect there are two strings of molecules, one centered at  $x=1/4, y=1/4$  and the other at  $x=3/4, y=3/4$  but the orientation along z of the one string with respect to the other was not yet fixed. Then too there is the question as to the orientation of the trapezoid of the chlorines in the symmetry plane. The projection does not reveal whether the long or short base is most advanced along z.

In an attempt to resolve these ambiguities, reflection amplitudes  $F(00\lambda)$  were calculated for motion of the molecule along z with a chosen orientation of the trapezoid. Since no agreement of the calculated amplitudes with those observed was obtained, the obvious step would have been to reverse the molecule and again compare amplitudes.

### Patterson Sections $P(0,Y,0)$ and $P(X,1/2,Z)$ .

A better idea presented itself however. If one calculates the Patterson section  $P(X,1/2,Z)$  he obtains peaks resulting from vectors whose Y separation is  $1/2$ . For atoms related to each other by the screw axis, peaks should appear in the section at  $2x, 1/2, 2z$ . Since x parameters are known, one would then be able to determine

## Orientation Along z.

At this stage, x and y coordinates were fairly well determined. The next problem was to determine positions along z. In effect there are two strings of molecules, one centered at  $x=1/2, y=1/2$  and the other at  $x=3/4, y=3/4$  but the orientation along z of the one string with respect to the other was not fixed. Then too there is the question as to the orientation of the triphenyl of the chlorines in the symmetry plane. The projection does not reveal whether the long or short axis is most advanced along z.

In an attempt to resolve these ambiguities, reflection amplitudes  $F(00\lambda)$  were calculated for motion of the molecule along z with a chosen orientation of the triphenyl. Since no agreement of the calculated amplitudes with those observed was obtained, the obvious step would have been to reverse the molecule and again compare amplitudes.

Triphenyl sections  $F(0, y, 0)$  and  $F(x, 1/2, z)$ .

A better idea presented itself however. If one calculates the triphenyl section  $F(x, 1/2, z)$  he obtains peaks resulting from vectors whose y separation is  $1/2$ . For atoms related to each other by the screw axis, peaks should appear in the section at  $2x, 1/2, 2z$ . Since x parameters are known, one would then be able to determine

z coordinates from peaks that are sufficiently resolved. There will of course be peaks indicating vectors between non-equivalent atoms lying in the symmetry planes.

The sections were made using relative  $|F_{hk\ell}|^2$  values divided by an arbitrary temperature factor  $e^{-3\left(\frac{\sin\theta}{\lambda}\right)^2}$  to sharpen up the peaks and to improve resolution. At the same time it was decided to compute the Patterson line section  $P(0,Y,0)$  which will give peaks corresponding to vectors which are parallel to the unique axis. For the molecule under discussion accurate separations of fourfold chlorine atoms should be observed as indeed they were.

The line section may be expressed as:

$$P(0,Y,0) = |F_{000}|^2 + 2 B_0 + 4 \sum_{k=1}^{\infty} B_k \cos 2 \pi k Y$$

$$\text{where } B_0 = 1/2 \sum_{h=1}^{\infty} |F_{h00}|^2 + 1/2 \sum_{h=-\infty}^{\infty} \sum_{\ell=1}^{\infty} |F_{h0\ell}|^2$$

$$B_k = 1/2 |F_{0k0}|^2 + \sum_{h=1}^{\infty} |F_{hk0}|^2 + \sum_{h=-\infty}^{\infty} \sum_{\ell=1}^{\infty} |F_{hk\ell}|^2$$

Figure 4 shows  $P(0,Y,0)$  for  $C_4Cl_8$ . It was only necessary to evaluate the function from  $Y=0$  to  $Y=1/2$  since the latter point is a center of symmetry.

The peak at  $Y=0$  arises from null vectors for each of the atoms in the unit cell. The peak at  $Y=0.325$  must be



There will be some 4-5 years between the time the first of these are put into service and the time the last of them are put into service.

The section was very noisy relative to other sections.

1. The first of these is the fact that the Commission has not yet received any information from the Government of the United States regarding the results of its investigation of the activities of the American Friends Service Committee in the Philippines.

Received 10/1/99; revised 10/1/99; accepted 10/1/99.

...the ... ..

There is a possibility that the observed

Instead they were:

The time taken may be expressed as:

$$|000\rangle = (0.5, 0.5)$$

$$S \left( \frac{1}{\lambda_{\text{eff}}} \right) \frac{1}{\lambda_{\text{eff}}} \sum_{\lambda=0}^{\infty} \frac{\lambda^{\lambda}}{\lambda!} \lambda! = S \left( \frac{1}{\lambda_{\text{eff}}} \right) \frac{1}{\lambda_{\text{eff}}} \sum_{\lambda=0}^{\infty} \frac{\lambda^{\lambda}}{\lambda!} \lambda! = 1$$

$$^{\circ} |\lambda_{2n}| \sum_{j=0}^{\infty} \frac{\lambda_j}{2^{j+1}} + ^{\circ} |\cos \theta| \left| \sum_{j=0}^{\infty} \frac{\lambda_j}{2^{j+1}} \right| + ^{\circ} |\cos \theta| \sqrt{2} \lambda_0$$

Figure 4 shows the results of the regression analysis. The results indicate that the model is significant at the 0.05 level,  $F(1, 10) = 10.00$ ,  $p < 0.05$ . The results also indicate that the model explains 45% of the variance in the dependent variable,  $R^2 = 0.45$ . The results further indicate that the model is a good fit for the data,  $F(1, 10) = 10.00$ ,  $p < 0.05$ . The results also indicate that the model is a good fit for the data,  $F(1, 10) = 10.00$ ,  $p < 0.05$ .

we evaluate the function  $f_{\text{eval}}(x)$  to yield value  $y$ .

where  $\rho$  is a constant of symmetry.

to date not verified; this could indicate they are being used

[illegible]



due to the shorter fourfold Cl-Cl spacing in the molecule.  
Thus if

$$1/2 - 2y = 0.325$$

$$\text{then } y = 0.083.$$

The peak at  $Y=1/2$  is associated with overlapping peaks resulting from the longer fourfold Cl-Cl spacing. If the single peak is resolved into the proper component peaks as shown by the dotted curves in the figure then

$$1/2 - 2y = 0.466$$

$$\text{and } y = 0.017.$$

The small hump at  $Y = 0.207$  may be a spurious peak resulting from the "sharpening up" process or may actually correspond to the expected fourfold carbon separation of  $2.2 \text{ \AA}$ . If

$$1/2 - 2y = 0.206$$

$$\text{then } y = 0.147$$

At any rate, good  $y$  parameters may now be assigned to the fourfold chlorines atoms.

Returning to the section  $P(X, 1/2, Z)$  it can be shown:

$$\begin{aligned} P(X, 1/2, Z) = & |F_{000}|^2 + 2B_{00} + 4 \sum_{h=1}^{\infty} B_{h0} \cos 2\pi hX \\ & + 4 \sum_{h=0}^{\infty} \sum_{\ell=1}^{\infty} B_{h\ell}^{11} \cos 2\pi hX \cos 2\pi \ell Z \\ & - 4 \sum_{h=0}^{\infty} \sum_{\ell=1}^{\infty} B_{h\ell}^{11} \sin 2\pi hX \sin 2\pi \ell Z \end{aligned}$$

for the vector function  $\mathbf{F}(\mathbf{r}, \mathbf{r}') = \mathbf{F}(\mathbf{r}, \mathbf{r}')$  in the volume.

Thus if

$$\mathbf{F}(\mathbf{r}, \mathbf{r}') = \mathbf{F}(\mathbf{r}, \mathbf{r}')$$

$$\mathbf{F}(\mathbf{r}, \mathbf{r}') = \mathbf{F}(\mathbf{r}, \mathbf{r}')$$

The peak of  $\mathbf{F}(\mathbf{r}, \mathbf{r}')$  is associated with overlapping peaks  
resulting from the angular function  $\mathbf{F}(\mathbf{r}, \mathbf{r}')$ . If the  
angular peak is removed from the angular component peaks  
as shown by the dashed curve in the figure then

$$\mathbf{F}(\mathbf{r}, \mathbf{r}') = \mathbf{F}(\mathbf{r}, \mathbf{r}')$$

$$\mathbf{F}(\mathbf{r}, \mathbf{r}') = \mathbf{F}(\mathbf{r}, \mathbf{r}')$$

The small curve at  $\mathbf{F}(\mathbf{r}, \mathbf{r}') = \mathbf{F}(\mathbf{r}, \mathbf{r}')$  may be a spurious peak resulting  
from the "removing" of peaks or may actually correspond  
to the expected function  $\mathbf{F}(\mathbf{r}, \mathbf{r}')$  at  $\mathbf{F}(\mathbf{r}, \mathbf{r}')$ . If

$$\mathbf{F}(\mathbf{r}, \mathbf{r}') = \mathbf{F}(\mathbf{r}, \mathbf{r}')$$

$$\mathbf{F}(\mathbf{r}, \mathbf{r}') = \mathbf{F}(\mathbf{r}, \mathbf{r}')$$

at any rate, good  $\mathbf{F}(\mathbf{r}, \mathbf{r}')$  patterns may now be assigned to the  
function  $\mathbf{F}(\mathbf{r}, \mathbf{r}')$ .

According to the equation  $\mathbf{F}(\mathbf{r}, \mathbf{r}') = \mathbf{F}(\mathbf{r}, \mathbf{r}')$  it can be shown

$$\mathbf{F}(\mathbf{r}, \mathbf{r}') = \mathbf{F}(\mathbf{r}, \mathbf{r}')$$

$$\mathbf{F}(\mathbf{r}, \mathbf{r}') = \mathbf{F}(\mathbf{r}, \mathbf{r}')$$

$$\mathbf{F}(\mathbf{r}, \mathbf{r}') = \mathbf{F}(\mathbf{r}, \mathbf{r}')$$

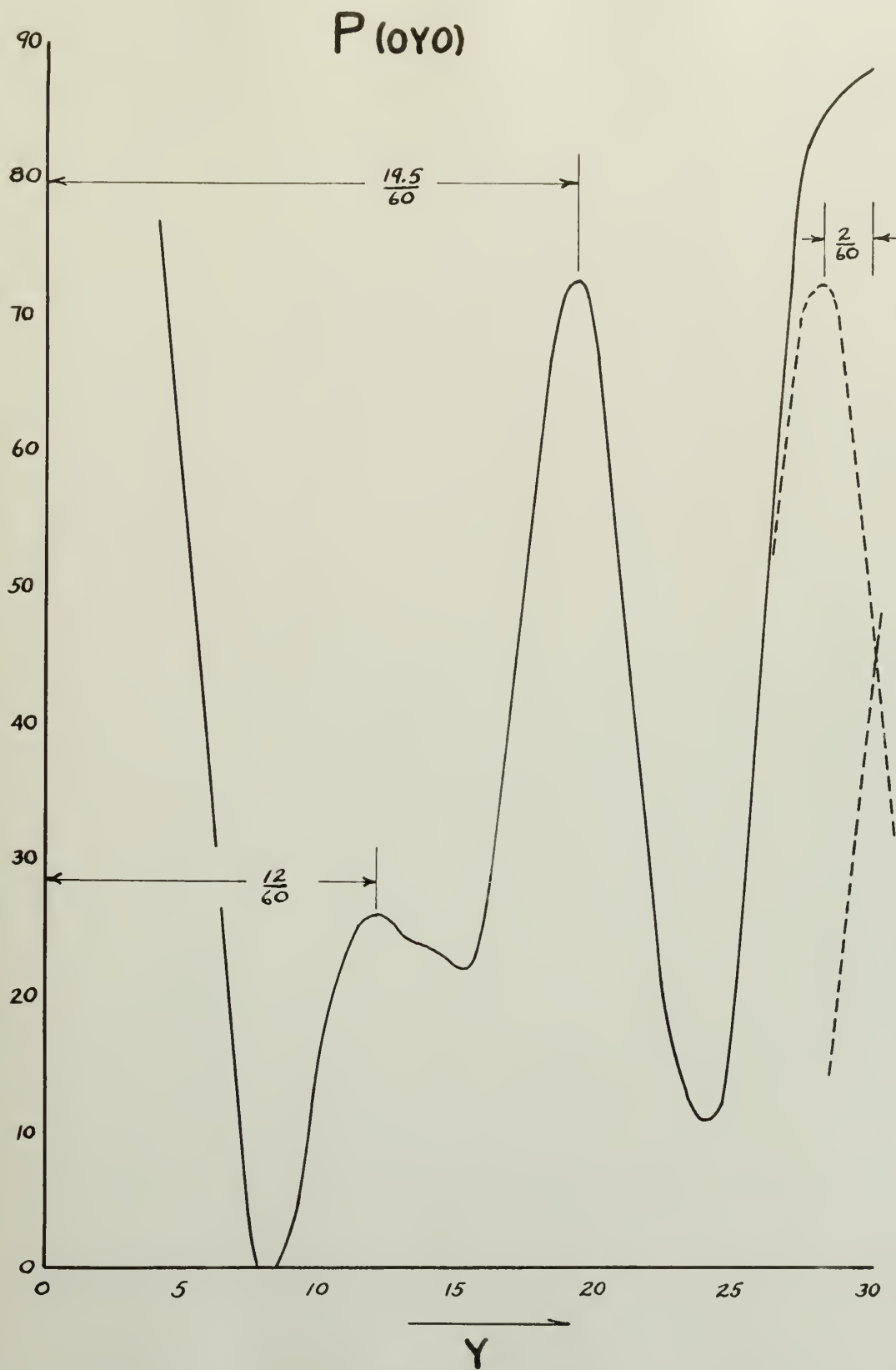
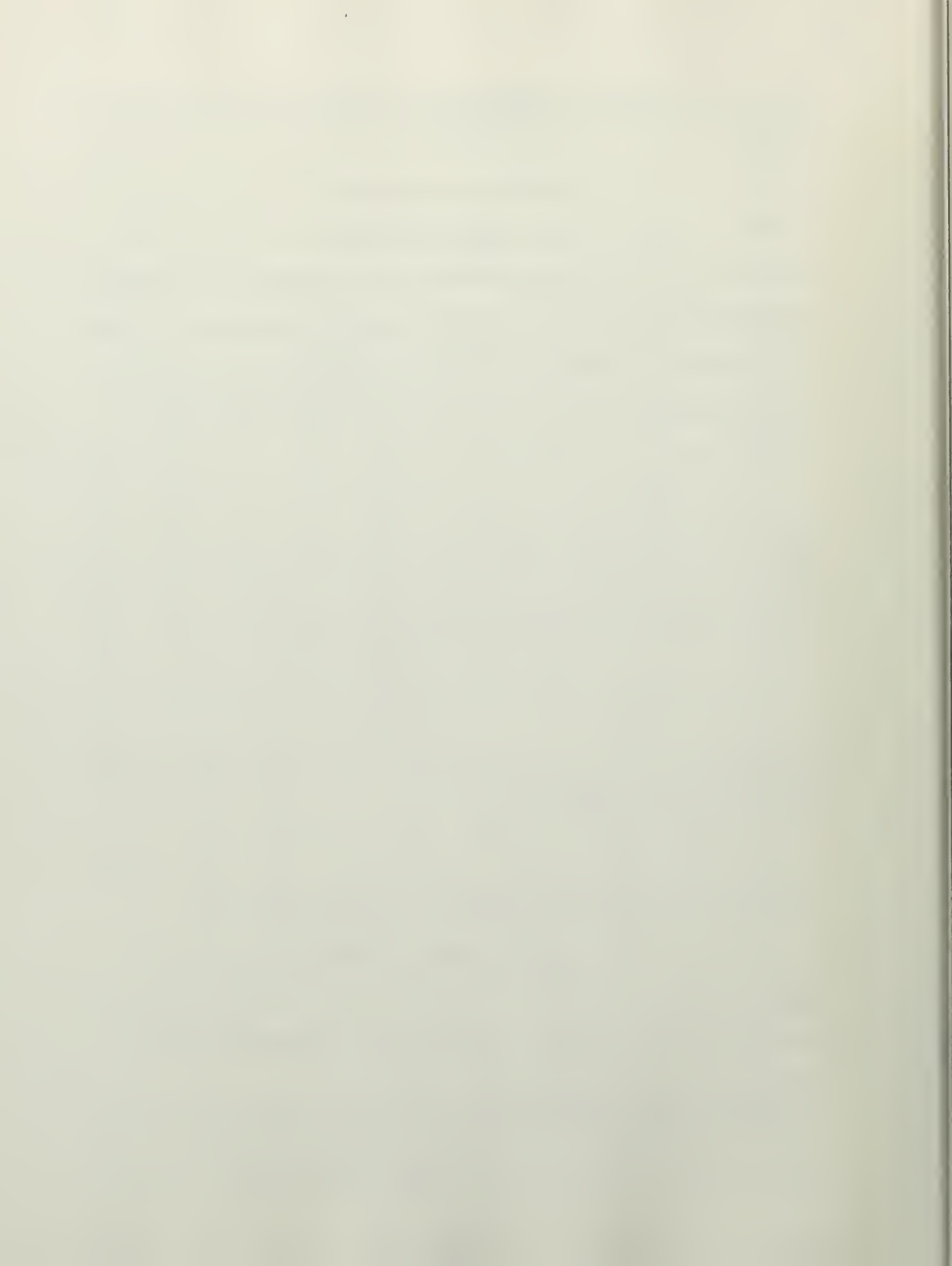


Figure 4





$$\text{where } B_{00} = 1/2 \sum_{k=2}^{\infty} |F_{0k0}|^2$$

$$B_{h0} = 1/2 |F_{h00}|^2 + \sum_{k=1}^{\infty} (-1)^k |F_{hk0}|^2$$

$$B^{1h\ell} = B_{h\ell} + B_{\bar{h}\ell}$$

$$B^{11h\ell} = B_{h\ell} - B_{\bar{h}\ell}$$

$$\text{and } B_{h\ell} = 1/2 |F_{h0\ell}|^2 + \sum_{k=1}^{\infty} (-1)^k |F_{hk\ell}|^2.$$

The section is shown in Figure 5. It is apparent that peaks indicative of vectors of  $X = 2x = 1/2$ , of which there will be many arising from the fourfold atoms, all run together so cannot be clearly resolved. However by considering the  $x$  parameters of the twofold chlorines (i.e. those in the symmetry plane) it was possible to pick the proper orientation of the trapezoid they form and to obtain their proper  $z$  coordinates. It was established that the short base of this trapezoid was most advanced in the  $+z$  direction and the proper orientation is as shown in Figure 2. Parameter values available now are listed. Small changes in  $x$  parameters have occurred as a result of including all the data in the summation.

$$|000| = \sqrt{2} \sum_{k=1}^{\infty} |F_{0k0}|^2 \quad \text{where}$$

$$|000| = \sqrt{2} |F_{000}|^2 + \sum_{k=1}^{\infty} (1-1)^2 |F_{0k0}|^2$$

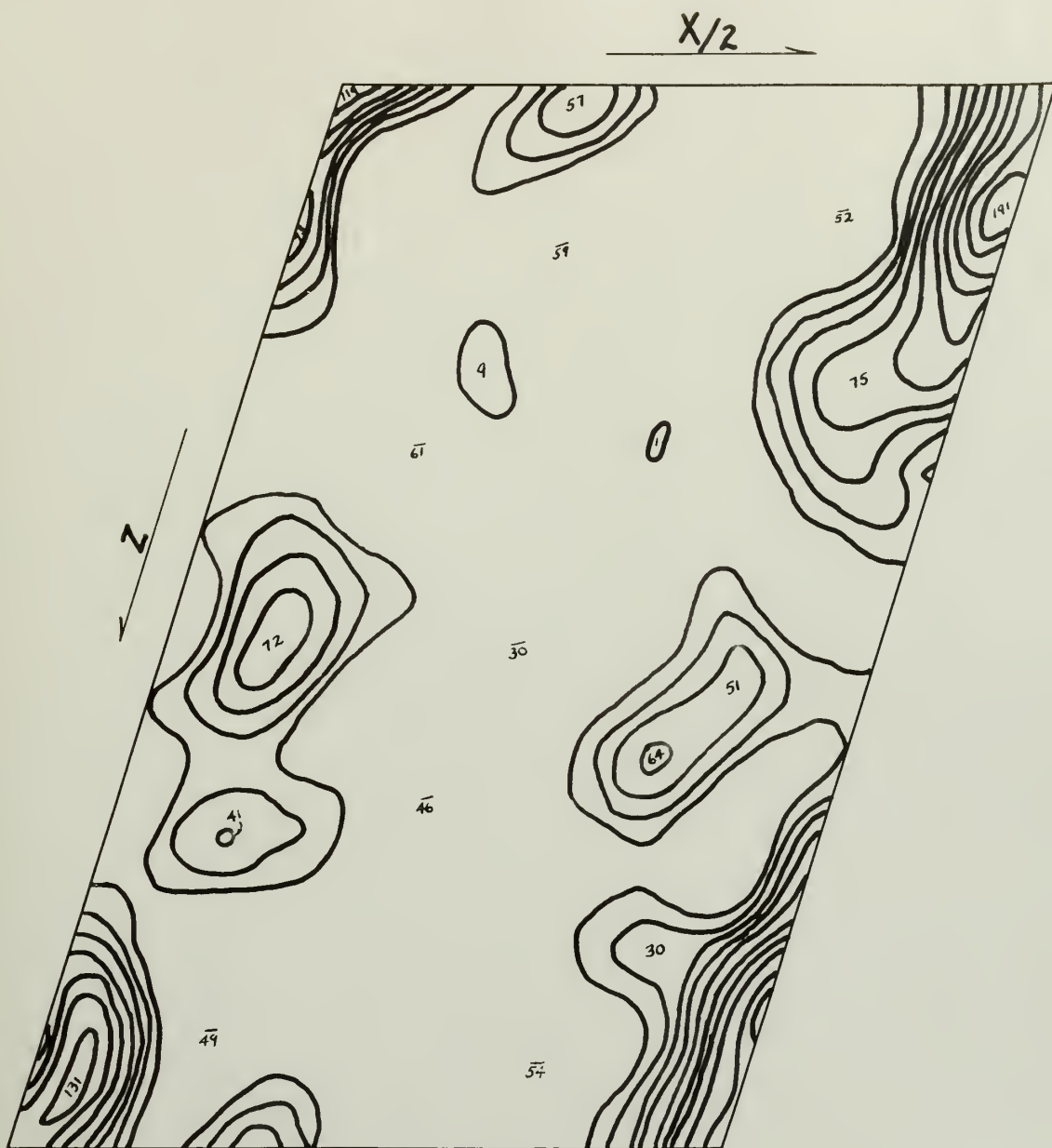
$$|110| = \sqrt{2} |F_{110}|^2 + \sum_{k=1}^{\infty} |F_{1k0}|^2$$

$$|111| = \sqrt{2} |F_{111}|^2 + \sum_{k=1}^{\infty} |F_{1k1}|^2$$

$$|112| = \sqrt{2} |F_{112}|^2 + \sum_{k=1}^{\infty} (1-1)^2 |F_{1k2}|^2 \quad \text{and}$$

The section is shown in Figure 2. It is apparent

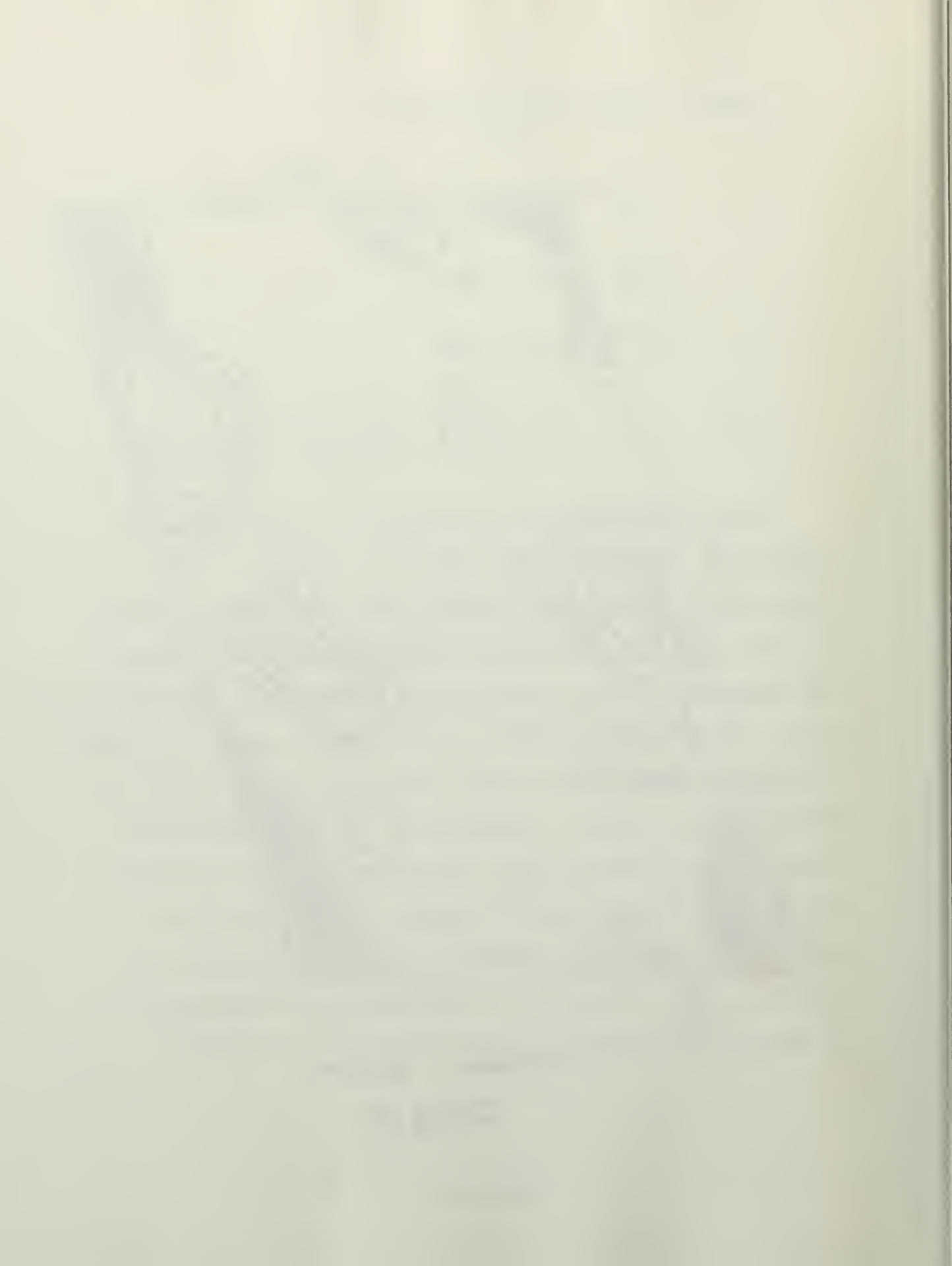
that peaks indicative of vectors of  $k = 2\pi/\lambda$  of which there will be many arising from the fourfold axes, all run together so cannot be easily resolved. However by considering the  $k$  parameters of the twofold chains (i.e. those in the symmetry plane) it was possible to pick the proper orientation of the tripole they form and to obtain their proper  $k$  coordinates. It was established that the short case of this tripole was most advanced in the  $z$  direction and the proper orientation is as shown in Figure 3. Parameter values available now are listed. Small changes in  $k$  parameters have occurred as a result of including all the data in the summation.



PATTERSON SECTION

$$P(x^{\frac{1}{2}}z)$$

Figure 5





	x	y	z
C <sub>1</sub>	+0.108	1/4	+0.238
C <sub>2</sub>	+0.250	+0.147	+0.230
C <sub>3</sub>	+0.392	1/4	+0.362
Cl <sub>1</sub>	+0.245	+0.088	-0.035
Cl <sub>4</sub>	+0.238	+0.017	+0.425
Cl <sub>5</sub>	+0.470	1/4	+0.655
Cl <sub>6</sub>	+0.033	1/4	+0.488
Cl <sub>7</sub>	-0.078	1/4	-0.017
Cl <sub>8</sub>	+0.568	1/4	+0.225

Fourier Projection  $\rho(x, z)$ .

In order to obtain better x and z coordinates the phases of the  $h0\ell$  reflections were calculated and the Fourier projection  $\rho(x, z)$  on (010) evaluated. Here

$$\begin{aligned}
 K \rho(x, z) = & F_{000} + \sum_{h=0}^{\infty} \sum_{\ell=1}^{\infty} (F_{h0\ell} + F_{\bar{h}0\ell}) \cos 2\pi h x \cos 2\pi \ell z \\
 & + \sum_{h=1}^{\infty} F_{h00} \cos 2\pi h x \\
 & - \sum_{h=1}^{\infty} \sum_{\ell=1}^{\infty} (F_{h0\ell} - F_{\bar{h}0\ell}) \sin 2\pi h x \sin 2\pi \ell z.
 \end{aligned}$$

There was some superposition of peaks corresponding to twofold chlorines in this projection and carbon maxima were rather fuzzy. The fourfold chlorines were well defined. Atomic coordinates were obtained, phases were redetermined,

	x	y	z
C1	+0.108	1/4	+0.123
C2	+0.120	+0.147	+0.130
C3	+0.132	1/4	+0.143
C4	+0.142	+0.158	+0.155
C5	+0.150	+0.167	+0.163
C6	+0.157	1/4	+0.168
C7	+0.163	1/4	+0.173
C8	+0.168	1/4	+0.177
C9	+0.173	1/4	+0.182

Fourier projection  $\rho(x, z)$ .

In order to obtain better x and z coordinates the phases of the  $h0\ell$  reflections were calculated and the Fourier projection  $\rho(x, z)$  on (000) evaluated, where

$$\rho(x, z) = 1000 + \sum_{h=0}^{\infty} \sum_{\ell=1}^{\infty} (F_{h0\ell} + F_{h0\ell}^*) \cos 2\pi h x \cos 2\pi \ell z$$

$$+ \sum_{h=1}^{\infty} \sum_{\ell=0}^{\infty} F_{h0\ell} \cos 2\pi h x$$

$$+ \sum_{h=1}^{\infty} \sum_{\ell=1}^{\infty} (F_{h0\ell} - F_{h0\ell}^*) \sin 2\pi h x \sin 2\pi \ell z.$$

There was some superposition of peaks corresponding to twofold chains in this projection and carbon atoms were rather fuzzy. The twofold chains were well defined. Atomic coordinates were obtained, phases were redetermined,

and the Fourier function re-evaluated until no sign changes occurred. Figure 6 shows the projection of matter in the unit cell along the unique axis. The overlap of the twofold chlorines is evident. Atomic coordinates are now:

	x	y	z
C <sub>1</sub>	+0.100	1/4	+0.233
C <sub>2</sub>	+0.238	+0.147	+0.262
C <sub>3</sub>	+0.378	1/4	+0.338
Cl <sub>1</sub>	+0.244	+0.088	-0.008
Cl <sub>4</sub>	+0.238	+0.017	+0.423
Cl <sub>5</sub>	+0.467	1/4	+0.636
Cl <sub>6</sub>	+0.012	1/4	+0.458
Cl <sub>7</sub>	-0.092	1/4	-0.003
Cl <sub>8</sub>	+0.572	1/4	+0.255

It may be well to note that coordinates given by the projections  $\rho(x,y)$  and  $\rho(x,z)$  cannot be considered as final. The X-ray photographs of the crystals show several reflections of considerable amplitude at high values of  $(\sin \theta)/\lambda$  indicating that the Fourier series will be slow in converging. To obtain the best set of positions all the data must be used and even then there may be errors due to non-convergence. This point will be discussed later.

and the Fourier transform re-evaluated with no sign changes occurred. Figure 4 shows the projection of the unit cell along the x-axis. The values of the Fourier transform are plotted. Atomic positions are not

$x$	$y$	$z$	$f$
0.100	0.100	0.100	0.100
0.100	0.100	0.100	0.100
0.100	0.100	0.100	0.100
0.100	0.100	0.100	0.100
0.100	0.100	0.100	0.100
0.100	0.100	0.100	0.100
0.100	0.100	0.100	0.100
0.100	0.100	0.100	0.100
0.100	0.100	0.100	0.100
0.100	0.100	0.100	0.100
0.100	0.100	0.100	0.100
0.100	0.100	0.100	0.100
0.100	0.100	0.100	0.100
0.100	0.100	0.100	0.100
0.100	0.100	0.100	0.100

It may be well to note that coordinates given by the projections  $\psi(x,y)$  and  $\phi(x,y)$  cannot be considered as final. The 2-ray photographs of the crystals show several reflections of considerable amplitude at high values of  $\psi$  and  $\phi$ . Indicating that the Fourier series will be slow in converging. To obtain the best set of positions all the data must be used and even then there may be errors due to non-convergence. This point will be discussed later.



$x$

$z$

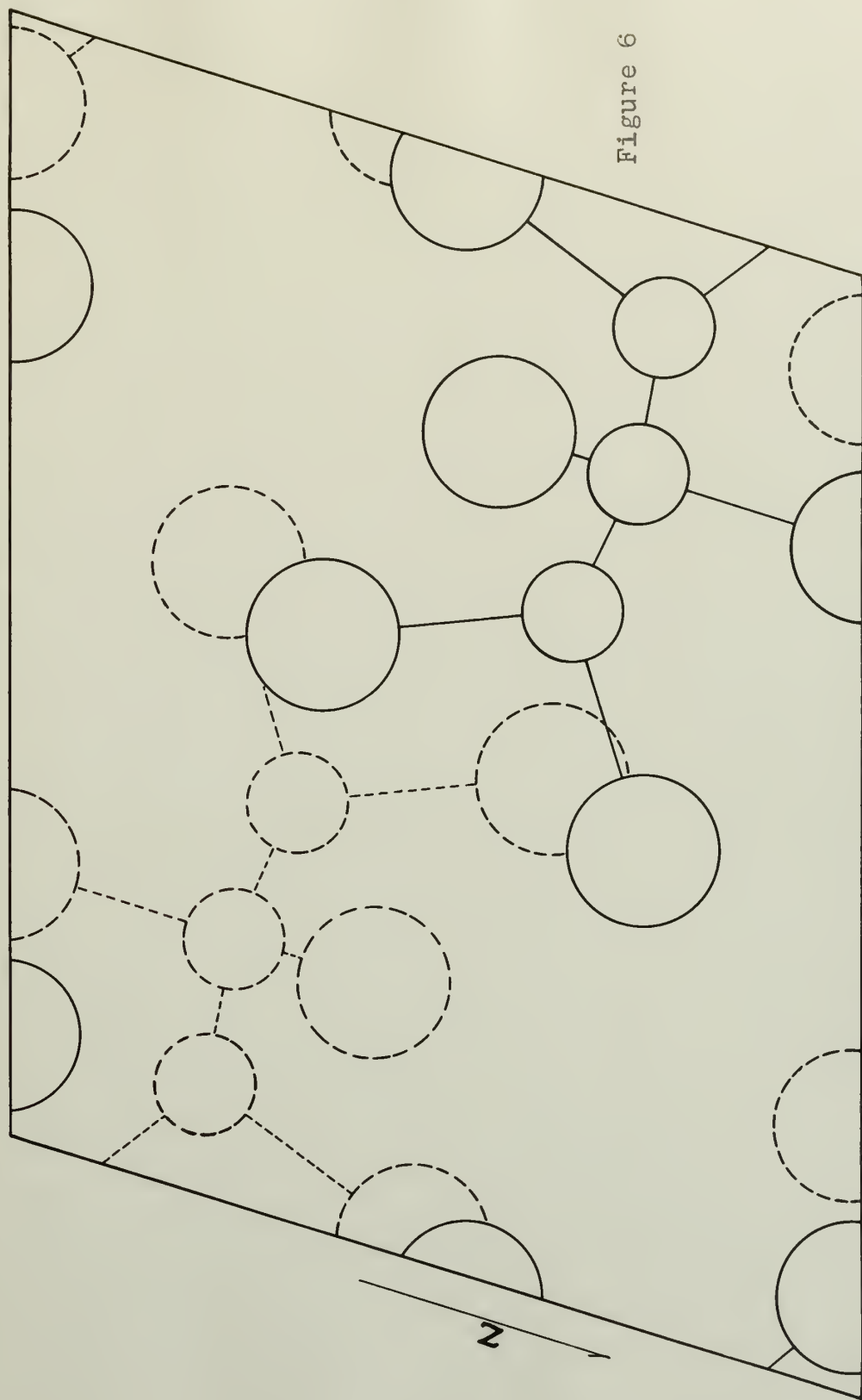


Figure 6

PROJECTION OF UNIT CELL ALONG UNIQUE AXIS



Fourier Sections  $\rho(x, 1/4, z)$  and  $\rho(1/4, y, z)$ .

It is particularly fortunate that half of the atoms of the molecule lie in the symmetry plane  $y = 1/4$  and even moreso that the other half lie almost exactly in the plane  $x = 1/4$ . Thus by taking sections through the unit cell at  $y = 1/4$  and at  $x = 1/4$  good parameter values should be obtained. This is particularly true since all reflections are used in the syntheses.

The Fourier section  $\rho(x, 1/4, z)$  may be evaluated as:

$$\begin{aligned}
 K\rho(x, 1/4, z) = & 4 \sum_{h=1}^{\infty} \sum_{\ell=1}^{\infty} A^1 \cos 2\pi hx \cos 2\pi \ell z \\
 & - 4 \sum_{h=1}^{\infty} \sum_{\ell=1}^{\infty} A^{11} \sin 2\pi hx \sin 2\pi \ell z \\
 & + 4 \sum_{\ell=1}^{\infty} A^{111} \cos 2\pi \ell z + 4 \sum_{h=1}^{\infty} A^{1111} \cos 2\pi hx \\
 & - 4 \sum_{h=1}^{\infty} \sum_{\ell=1}^{\infty} B^1 \cos 2\pi hx \sin 2\pi \ell z \\
 & - 4 \sum_{h=1}^{\infty} \sum_{\ell=1}^{\infty} B^{11} \sin 2\pi hx \cos 2\pi \ell z \\
 & - 4 \sum_{h=1}^{\infty} B^{111} \sin 2\pi hx - 4 \sum_{\ell=1}^{\infty} B^{1111} \sin 2\pi \ell z,
 \end{aligned}$$

(constant additive terms are neglected) where:

$$A^1 = 1/2 (F_{h0\ell} + F_{\bar{h}0\ell}) + \sum_{\substack{k=2n \\ n \geq 1}}^{\infty} (F_{hk\ell} + F_{\bar{h}k\ell}) (-1)^{k/2}.$$

Fourier series  $\psi(x, y, z)$  and  $\psi(x, y, z)$ .

It is particularly fortunate that half of the atoms of the molecule lie in the symmetry plane  $y = 1/4$  and even more so that the other half lie almost exactly in the plane  $x = 1/4$ . Thus by taking sections through the unit cell at  $y = 1/4$  and at  $x = 1/4$  good parameter values should be obtained. This is particularly true since all reflections are used in the synthesis.

The Fourier section  $\psi(x, 1/4, z)$  may be evaluated as

$$\psi(x, 1/4, z) = \sum_{h=1}^{\infty} \sum_{k=1}^{\infty} A_{hk} \cos 2\pi h x \cos 2\pi k z$$

$$= \sum_{h=1}^{\infty} \sum_{k=1}^{\infty} A_{hk} \sin 2\pi h x \sin 2\pi k z$$

$$= \sum_{h=1}^{\infty} \sum_{k=1}^{\infty} A_{hk} \cos 2\pi h x \sin 2\pi k z$$

$$= \sum_{h=1}^{\infty} \sum_{k=1}^{\infty} A_{hk} \sin 2\pi h x \cos 2\pi k z$$

$$= \sum_{h=1}^{\infty} \sum_{k=1}^{\infty} A_{hk} \sin 2\pi h x \sin 2\pi k z$$

$$= \sum_{h=1}^{\infty} \sum_{k=1}^{\infty} A_{hk} \cos 2\pi h x \cos 2\pi k z$$

(constant additive terms are neglected) where

$$A_{hk} = \sum_{n=1}^{\infty} \psi(x, y, z) \cos 2\pi n x \cos 2\pi n y \cos 2\pi n z$$



$$A^{11} = 1/2 (F_{h0l} - F_{\bar{h}0l}) + \sum_{\substack{k=2n \\ n \geq 1}}^{\infty} (F_{hkl} - F_{\bar{h}kl}) (-1)^{k/2},$$

$$A^{111} = 1/2 F_{00l} + \sum_{\substack{k=2n \\ n \geq 1}}^{\infty} F_{0kl} (-1)^{k/2},$$

$$A^{1111} = 1/2 F_{h00} + \sum_{\substack{k=2n \\ n \geq 1}}^{\infty} F_{hk0} (-1)^{k/2},$$

$$B^1 = \sum_{\substack{k=2n+1 \\ n \geq 0}}^{\infty} (F_{hkl} + F_{\bar{h}kl}) \left( \sin \frac{\pi k}{2} \right),$$

$$B^{11} = \sum_{\substack{k=2n+1 \\ n \geq 0}}^{\infty} (F_{hkl} - F_{\bar{h}kl}) \left( \sin \frac{\pi k}{2} \right),$$

$$B^{111} = \sum_{\substack{k=2n+1 \\ n \geq 0}}^{\infty} F_{hk0} \left( \sin \frac{\pi k}{2} \right),$$

$$B^{1111} = \sum_{\substack{k=2n+1 \\ n \geq 0}}^{\infty} F_{0kl} \left( \sin \frac{\pi k}{2} \right).$$

The section in the plane  $x = 1/4$  may be evaluated as (neglecting constant additive terms):

$$K\rho(1/4, y, z) = 4 \sum_{\substack{k=2n \\ n \geq 0}}^{\infty} \sum_{l=1}^{\infty} \left[ \sum_{\substack{h=2n \\ n \geq 1}}^{\infty} (F_{hkl} + F_{\bar{h}kl}) \cos \frac{\pi h}{2} \right.$$

$$\left. + F_{0kl} \right] \cos 2\pi ky \cos 2\pi lz$$

$$+ 4 \sum_{\substack{k=2n \\ n \geq 0}}^{\infty} \left[ \sum_{\substack{h=2n \\ n \geq 0}}^{\infty} F_{hk0} \cos \frac{\pi h}{2} + 1/2 F_{0k0} \right] \cos 2\pi ky$$

$$I_1 = \sum_{n=0}^{\infty} \frac{1}{n!} \left( \frac{1}{2} \right)^n = e^{1/2}$$

$$I_2 = \sum_{n=0}^{\infty} \frac{1}{n!} \left( \frac{1}{2} \right)^n = e^{1/2}$$

$$I_3 = \sum_{n=0}^{\infty} \frac{1}{n!} \left( \frac{1}{2} \right)^n = e^{1/2}$$

$$I_4 = \sum_{n=0}^{\infty} \frac{1}{n!} \left( \frac{1}{2} \right)^n = e^{1/2}$$

$$I_5 = \sum_{n=0}^{\infty} \frac{1}{n!} \left( \frac{1}{2} \right)^n = e^{1/2}$$

$$I_6 = \sum_{n=0}^{\infty} \frac{1}{n!} \left( \frac{1}{2} \right)^n = e^{1/2}$$

$$I_7 = \sum_{n=0}^{\infty} \frac{1}{n!} \left( \frac{1}{2} \right)^n = e^{1/2}$$

The motion in the plane  $x = 1/2$  may be evaluated

as (neglecting constant additive terms):

$$H(\lambda, \gamma, z) = \sum_{n=0}^{\infty} \frac{1}{n!} \left( \frac{1}{2} \right)^n \cos \frac{\pi}{2} n$$

$$H(\lambda, \gamma, z) = \sum_{n=0}^{\infty} \frac{1}{n!} \left( \frac{1}{2} \right)^n \cos \frac{\pi}{2} n$$

$$H(\lambda, \gamma, z) = \sum_{n=0}^{\infty} \frac{1}{n!} \left( \frac{1}{2} \right)^n \cos \frac{\pi}{2} n$$

$$\begin{aligned}
& + 4 \sum_{\ell=1}^{\infty} \left[ \sum_{\substack{h=2n \\ n \geq 1}}^{\infty} \left( \frac{F_{h0\ell} + F_{\bar{h}0\ell}}{2} \right) \cos \frac{\pi h}{2} + 1/2 F_{00\ell} \right] \cos 2\pi \ell z \\
& - 4 \sum_{\substack{k=2n+1 \\ n \geq 0}}^{\infty} \sum_{\ell=1}^{\infty} \left[ F_{0k\ell} + \sum_{\substack{h=2n \\ n \geq 1}}^{\infty} (F_{hk\ell} + F_{\bar{h}k\ell}) \cos \frac{\pi h}{2} \right] \sin 2\pi ky \sin 2\pi \ell z \\
& - 4 \sum_{\substack{k=2n \\ n \geq 0}}^{\infty} \sum_{\ell=1}^{\infty} \left[ \sum_{\substack{h=2n+1 \\ n \geq 0}}^{\infty} (F_{hk\ell} - F_{\bar{h}k\ell}) \sin \frac{\pi h}{2} \right. \\
& \left. + \sum_{\substack{h=2n+1 \\ n \geq 0}}^{\infty} \left( \frac{F_{h0\ell} - F_{\bar{h}0\ell}}{2} \right) \sin \frac{\pi h}{2} \right] \cos 2\pi ky \sin 2\pi \ell z \\
& - 4 \sum_{\substack{k=2n+1 \\ n \geq 0}}^{\infty} \sum_{\ell=1}^{\infty} \left[ \sum_{\substack{h=2n+1 \\ n \geq 0}}^{\infty} (F_{hk\ell} - F_{\bar{h}k\ell}) \sin \frac{\pi h}{2} \right. \\
& \left. + \sum_{\substack{h=2n+1 \\ n \geq 0}}^{\infty} F_{hk0} \left( \sin \frac{\pi h}{2} \right) \right] \sin 2\pi ky \cos 2\pi \ell z.
\end{aligned}$$

The two Fourier functions were evaluated and appropriate parameters obtained. Phases were recalculated and the final sections are shown in Figures 7 and 8. Contours commence at 25 and are drawn at intervals of 25 units of arbitrary relative electron density. It is readily seen that the carbon positions as given by the weak carbon peaks will not be very accurate. The contours for all atoms were redrawn on large coordinate paper and final atomic coordinates obtained in this study are:

$$a_n = \frac{1}{\pi} \int_{-\pi}^{\pi} f(x) \cos nx \, dx$$

$$b_n = \frac{1}{\pi} \int_{-\pi}^{\pi} f(x) \sin nx \, dx$$

$$f(x) = \frac{1}{2} (f(x) + f(x))$$

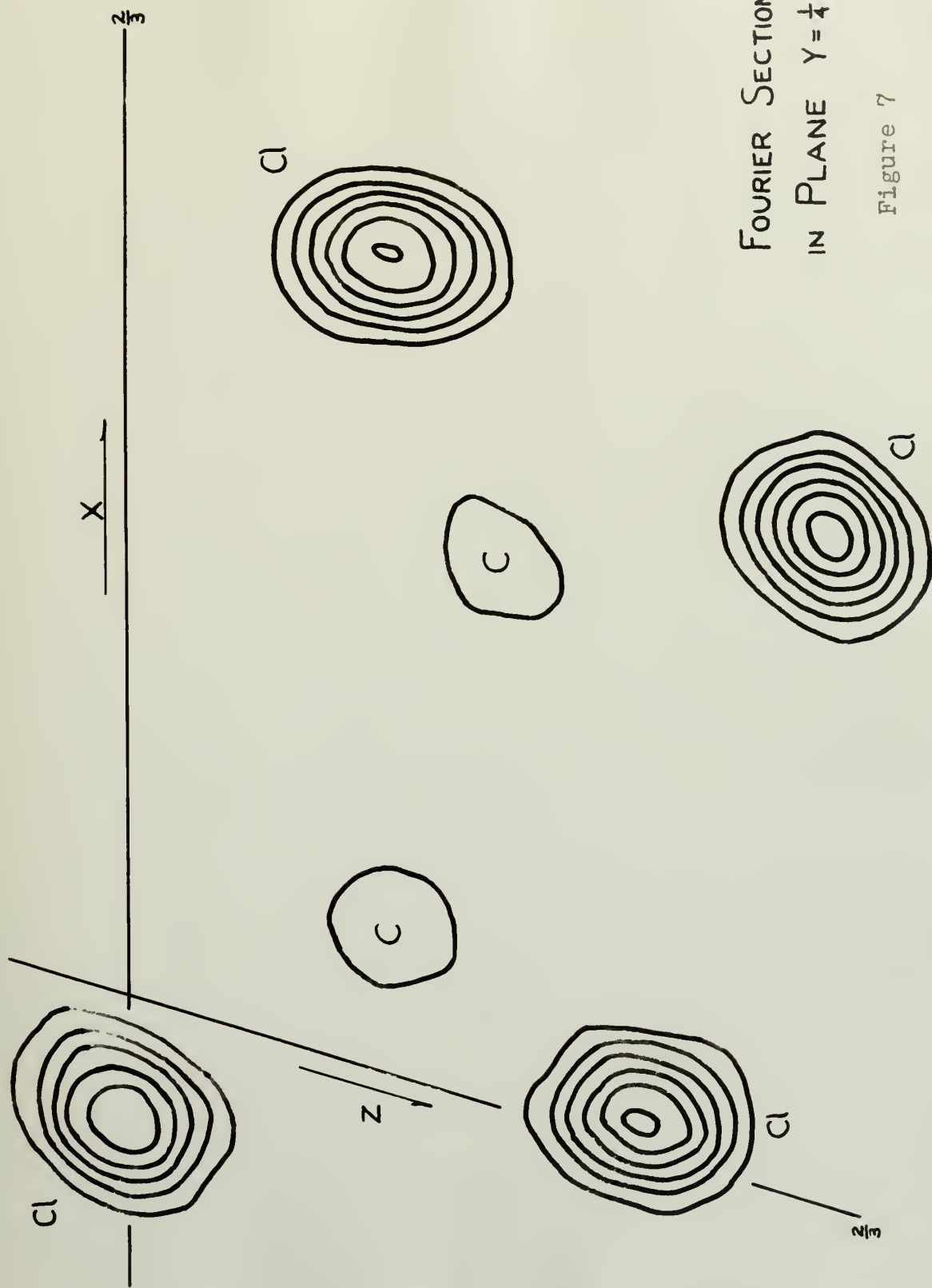
$$f(x) = \frac{1}{2} (f(x) + f(x))$$

$$f(x) = \frac{1}{2} (f(x) + f(x))$$

$$f(x) = \frac{1}{2} (f(x) + f(x))$$

The Fourier series were calculated and approx-  
imate derivatives obtained. These were recalculated and  
the final sections are shown in Figures 7 and 8. Calculations  
commenced at 10 and are given at intervals of 10 units of  
arbitrary relative electron density. It is readily seen  
that the carbon positions are given by the well defined  
peaks will not be very accurate. The constants for all  
atoms were taken as large constants paper and final  
atomic coordinates obtained in this study are:

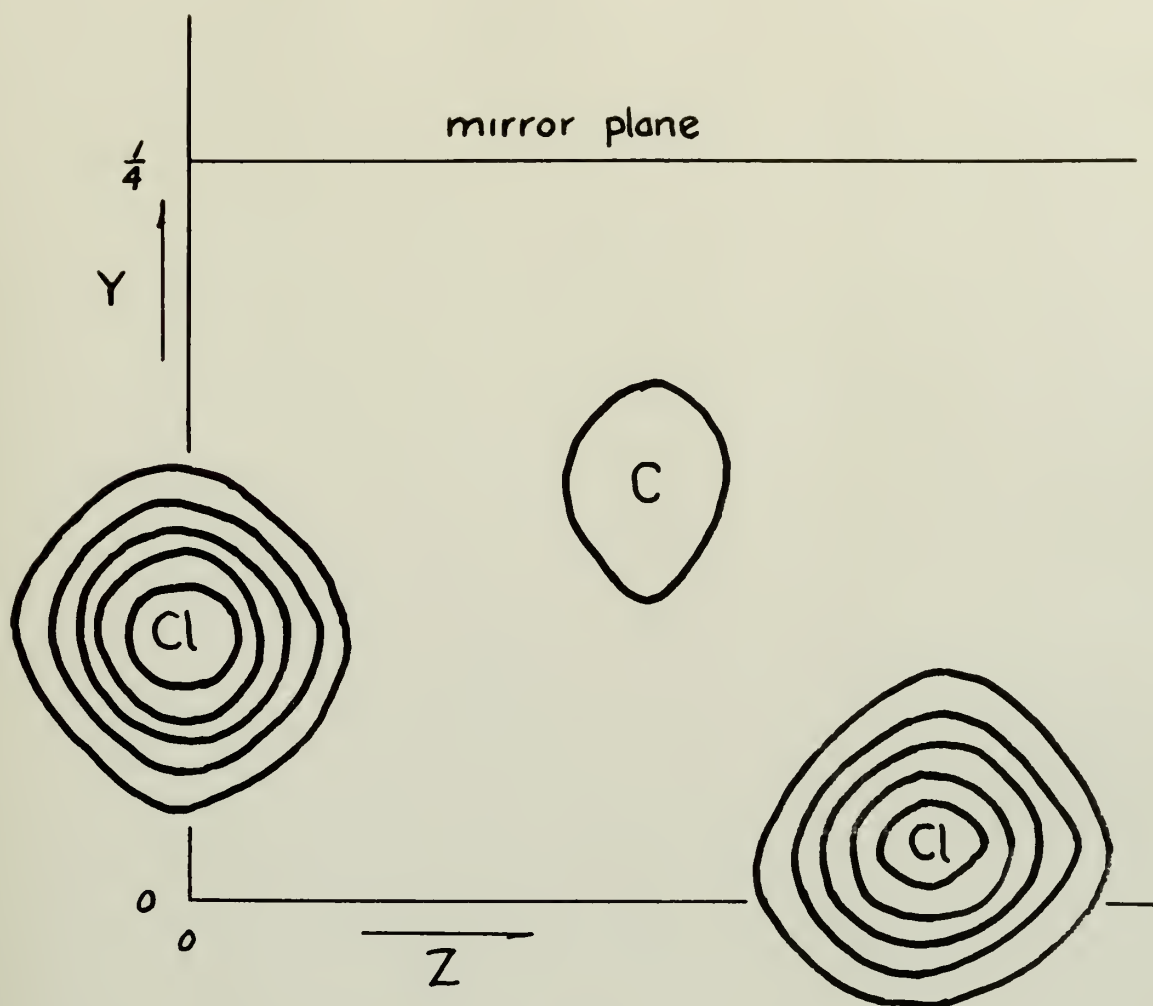




FOURIER SECTION  
IN PLANE  $Y = \frac{1}{4}$

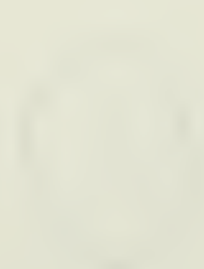
Figure 7





FOURIER SECTION IN PLANE  $x = \frac{1}{4}$

Figure 8



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	x	y	z
C <sub>1</sub>	+0.100	1/4	+0.246
C <sub>2</sub>	+0.242	+0.146	+0.264
C <sub>3</sub>	+0.378	1/4	+0.334
C <sub>11</sub>	+0.243	+0.091	-0.005
C <sub>14</sub>	+0.237	+0.019	+0.425
C <sub>15</sub>	+0.462	1/4	+0.638
C <sub>16</sub>	+0.022	1/4	+0.465
C <sub>17</sub>	-0.084	1/4	-0.006
C <sub>18</sub>	+0.564	1/4	+0.242

z	y	x	
590.0+	1\1	591.0+	10
580.0+	581.0+	580.0+	20
570.0+	1\1	570.0+	30
560.0-	190.0+	560.0+	40
550.0+	510.0+	550.0+	50
540.0+	1\1	540.0+	60
530.0+	1\1	530.0+	70
520.0-	1\1	520.0-	80
510.0+	1\1	510.0+	90

## COMPARISON OF CALCULATED AND OBSERVED AMPLITUDES

In order to compare calculated and observed amplitudes as a check on the validity of parameters obtained, the effects of thermal motion of the atoms must be considered. While not completely satisfactory an isotropic temperature factor of the form  $e^{-B \left( \frac{\sin \theta}{\lambda} \right)^2}$  (where B must be determined) may be used to multiply the amplitudes calculated on the assumption the atoms are at rest in order that a comparison may be made. This is the usual practice in X-ray work. In addition to temperature factor determination it must be remembered that observed amplitudes are on a relative basis. Thus a factor must be determined which will correct them to an absolute scale.

Mathematically then:

$$K |F_{Obs.}| = |F_{calc}| e^{-B \left( \frac{\sin \theta}{\lambda} \right)^2}.$$

This may be rewritten in logarithmic form as

$$\ln \frac{|F_{calc.}|}{|F_{obs.}|} = \ln K + B \left( \frac{\sin \theta}{\lambda} \right)^2$$

which is of the form  $y=mx+b$ , an equation of a straight line where  $b=\ln K$  and  $m=B$ . The method of least squares was used to determine best values of the slope and intercept of the straight line. It was found that

$$K = 2.87$$

$$B = 2.97 \text{ \AA}^2$$

In order to compare calculated and observed results

as a check on the validity of parameters obtained, the effects of thermal motion of the atoms must be considered. While not completely satisfactory as a check, the calculation of the form factor  $f_0$  (where  $f_0$  is the calculated value) may be used to multiply the calculated values as the temperature of the atoms is at room temperature. This is the usual practice in X-ray work. In addition to temperature factor, the calculation is most complicated that observed results are on a relative basis. Thus a factor must be determined which will correct them to an absolute scale.

Mathematically then:

$$K |F_{obs}| = |F_{calc}| = \left| \sum_j f_j \exp(i 2\pi \mathbf{h} \cdot \mathbf{r}_j) \right|$$

This may be rewritten in logarithmic form as

$$\ln |F_{obs}| = \ln \left| \sum_j f_j \exp(i 2\pi \mathbf{h} \cdot \mathbf{r}_j) \right|$$

which is of the form  $y = ax + b$ , an equation of a straight line where  $y$  and  $x$  are. The method of least squares was used to determine best values of the slope and intercept of the straight line. It was found that

$$K = 2.27$$

$$b = 2.97$$



when the 592 forms were used in the calculation.

Table I lists  $\frac{(F_{\text{calc.}})}{2} e^{-3\left(\frac{\sin \theta}{\lambda}\right)^2}$  and  $2.87 F_{\text{obs.}}$

for each reflection;

TABLE I

$\frac{F_{hkl}}{2}$			$\frac{F_{hkl}}{2}$		
hkl	Calc.	Obs.	hkl	Calc.	Obs.
020	-29.0	35.0	300	- 9.5	11.5
040	+47.6	53.9	310	+21.6	25.0
060	-34.1	37.6	320	- 4.2	4.0
080	+30.4	31.5	330	+20.6	24.1
0,10,0	- 9.8	4.9	340	- 2.5	0
0,12,0	+16.7	18.3	350	+11.9	12.3
			360	- 0.2	0
100	+ 4.2	4.6	370	- 3.1	0
110	-25.5	28.4	380	- 1.9	0
120	+ 2.8	3.4	390	+ 3.3	0
130	-36.6	32.7	3,10,0	- 0.7	0
140	+ 0.5	0	3,11,0	+ 3.2	2.9
150	- 8.3	11.5	3,12,0	- 0.9	0
160	+0.5	0	3,13,0	+ 7.1	6.0
170	- 1.1	0			
180	+ 0.4	0	400	+38.1	45.6
190	- 0.3	0	410	+ 3.1	3.7
1,10,0	+ 0.5	0	420	+16.3	18.1
1,11,0	- 6.2	6.6	430	+ 4.0	4.6
1,12,0	+ 0.2	0	440	+ 2.6	5.2
1,13,0	- 6.6	9.5	450	+ 2.5	0
			460	- 2.5	0
200	-18.6	18.7	470	- 0.3	0
210	+ 2.2	2.3	480	+ 6.0	7.2
220	-68.2	57.5	490	+ 1.8	0
230	- 6.8	8.6	4,10,0	+ 4.3	4.6
240	+27.9	32.1	4,11,0	+ 1.3	0
250	+ 1.4	0	4,12,0	+ 4.5	4.3
260	-20.2	24.1			
270	- 8.8	0	500	+ 8.2	9.5
280	+ 9.8	8.6	510	-10.7	10.6
290	+ 0.5	0	520	+ 6.2	4.9
2,10,0	-18.2	20.7	530	-15.8	16.3
2,11,0	- 1.9	0	540	+ 1.4	0
2,12,0	+ 3.0	0	550	- 5.7	5.5
2,13,0	- 0.2	0	560	+ 1.4	0

where the 75% value was used in the calculation.

$$= \frac{1}{2} \left( \frac{1}{1} + \frac{1}{1} \right) = 1$$

1000 700 600 500 400 300 200 100 0

**ACKNOWLEDGMENTS**

[illegible]

TABLE I (Continued)

hk $\ell$	Calc.	Obs.	hk $\ell$	Calc.	Obs.
570	+ 0.4	0	950	- 7.8	8.0
580	+ 1.4	0	001	- 7.9	10.0
590	- 0.7	0	011	-22.9	22.4
5,10,0	+ 1.5	0	021	- 2.1	1.6
5,11,0	- 3.3	0	031	+ 5.4	6.6
600	-26.7	31.0	041	-35.0	35.6
610	- 7.0	6.0	051	- 6.8	5.2
620	- 6.3	4.6	061	-18.3	18.1
630	- 1.9	0	071	- 0.6	0
640	- 4.7	6.3	081	-10.5	10.6
650	- 4.2	4.0	091	- 9.8	8.3
660	+ 5.1	4.6	0,10,1	+ 6.4	0
670	+ 1.7	0	0,11,1	+ 0.7	0
680	- 6.1	7.2	0,12,1	+ 1.8	0
690	- 2.4	0	0,13,1	- 3.0	0
6,10,0	- 1.5	0	101	-10.9	6.6
6,11,0	+ 0.8	0	111	- 1.0	2.3
700	- 4.9	2.9	121	-19.5	21.2
710	- 0.3	0	131	-23.2	25.0
720	- 7.4	6.0	141	+ 2.0	2.3
730	+15.5	13.2	151	+ 9.9	7.8
740	+ 0.8	0	161	-14.3	14.4
750	- 2.0	0	171	+11.9	11.2
760	- 2.8	4.6	181	- 3.6	0
770	+ 4.2	0	191	+17.8	16.1
780	- 0.1	0	1,10,1	- 2.5	2.9
790	- 2.9	0	1,11,1	+ 2.4	0
800	+ 9.4	7.2	1,12,1	+ 2.1	0
810	+ 0.4	0	1,13,1	+ 1.4	0
820	+ 9.4	9.5	I01	+28.0	32.8
830	+ 5.0	5.5	I11	- 2.5	0
840	- 3.0	4.0	I21	+ 2.6	2.9
850	+ 0.4	0	I31	+26.6	26.2
860	+ 1.4	0	I41	+ 3.8	6.9
870	+ 1.0	0	I51	-11.8	9.2
880	+0	0	I61	+ 3.7	4.0
900	+ 9.9	10.9	I71	-12.3	11.5
910	-10.8	13.8	I81	+ 5.5	4.6
920	- 0.7	3.4	I91	-19.8	16.6
930	+ 1.7	4.6	I,10,1	- 1.7	0
940	+ 4.8	5.7	I,11,1	- 2.5	0
			I,12,1	+ 0.9	0



[illegible]



TABLE I (Continued)

hk $\lambda$	Calc.	Obs.	hk $\lambda$	Calc.	Obs.
<u>1</u> ,13,1	- 2.1	0	<u>3</u> 01	-27.6	33.0
201	-29.2	30.8	<u>3</u> 11	-33.2	31.6
211	+ 8.7	8.9	<u>3</u> 21	+ 9.9	8.9
221	+29.5	34.7	<u>3</u> 31	+10.4	9.2
231	- 0.2	0	<u>3</u> 41	-10.7	10.8
241	+ 4.5	5.5	<u>3</u> 51	-13.8	12.9
251	+ 3.6	0	<u>3</u> 61	+ 6.4	4.6
261	+33.8	30.1	<u>3</u> 71	+29.2	24.4
271	+ 3.9	0	<u>3</u> 81	- 9.4	5.7
281	- 5.0	0	<u>3</u> 91	+ 4.6	0
291	+ 8.5	6.6	<u>3</u> ,10,1	+ 5.4	0
2,10,1	+ 4.0	0	<u>3</u> ,11,1	+11.5	9.8
2,11,1	+ 1.5	0	<u>3</u> ,12,1	- 2.7	0
2,12,1	- 8.7	4.6	<u>3</u> ,13,1	- 4.4	0
2,13,1	+ 1.9	0			
<u>2</u> 01	+ 2.3	0	401	-20.2	28.1
<u>2</u> 11	- 4.4	4.0	411	+ 7.9	10.6
<u>2</u> 21	+ 9.5	9.2	421	+20.2	25.5
<u>2</u> 31	+15.7	16.4	431	-11.0	11.8
<u>2</u> 41	+28.2	30.4	441	-35.2	40.2
<u>2</u> 51	- 8.3	7.5	451	+ 6.3	8.9
<u>2</u> 61	+21.8	20.6	461	+ 3.1	4.0
<u>2</u> 71	+ 8.0	7.8	471	-12.3	11.2
<u>2</u> 81	+7.9	8.3	481	-17.8	14.4
<u>2</u> 91	- 0.3	3.7	491	- 2.1	0
<u>2</u> ,10,1	- 4.4	0	4,10,1	+12.1	8.9
<u>2</u> ,11,1	+ 3.9	0	4,11,1	- 6.0	5.2
<u>2</u> ,12,1	- 3.0	0	4,12,1	- 3.9	0
<u>2</u> ,13,1	- 1.2	0			
301	+16.7	15.8	<u>4</u> 01	+ 1.5	0
311	+20.4	25.6	<u>4</u> 11	- 5.4	5.7
321	+ 5.5	4.9	<u>4</u> 21	-10.8	11.5
331	+ 0.5	0	<u>4</u> 31	- 3.3	4.0
341	+ 9.7	9.8	<u>4</u> 41	-18.5	16.6
351	+ 6.7	6.9	<u>4</u> 51	+ 0.6	0
361	+ 6.5	8.0	<u>4</u> 61	-20.0	18.7
371	-19.7	17.8	<u>4</u> 71	+ 0.2	0
381	+ 6.6	5.7	<u>4</u> 81	- 5.0	4.0
391	- 6.1	6.6	491	- 1.9	0
3,10,1	- 2.0	0	4,10,1	+ 2.3	0
3,11,1	- 7.2	8.9	4,11,1	+ 0.1	0
3,12,1	+ 0.5	0	4,12,1	+ 3.5	0

## TABLE 1 (continued)

Code	Code	Code	Code	Code	Code
C.11	A.11	100	0	1.1	1.1
C.12	C.12	110	A.12	1.12	1.12
C.13	C.13	120	C.13	1.13	1.13
C.14	C.14	130	C.14	1.14	1.14
C.15	C.15	140	C.15	1.15	1.15
C.16	C.16	150	C.16	1.16	1.16
C.17	C.17	160	C.17	1.17	1.17
C.18	C.18	170	C.18	1.18	1.18
C.19	C.19	180	C.19	1.19	1.19
C.20	C.20	190	C.20	1.20	1.20
C.21	C.21	200	C.21	1.21	1.21
C.22	C.22	210	C.22	1.22	1.22
C.23	C.23	220	C.23	1.23	1.23
C.24	C.24	230	C.24	1.24	1.24
C.25	C.25	240	C.25	1.25	1.25
C.26	C.26	250	C.26	1.26	1.26
C.27	C.27	260	C.27	1.27	1.27
C.28	C.28	270	C.28	1.28	1.28
C.29	C.29	280	C.29	1.29	1.29
C.30	C.30	290	C.30	1.30	1.30
C.31	C.31	300	C.31	1.31	1.31
C.32	C.32	310	C.32	1.32	1.32
C.33	C.33	320	C.33	1.33	1.33
C.34	C.34	330	C.34	1.34	1.34
C.35	C.35	340	C.35	1.35	1.35
C.36	C.36	350	C.36	1.36	1.36
C.37	C.37	360	C.37	1.37	1.37
C.38	C.38	370	C.38	1.38	1.38
C.39	C.39	380	C.39	1.39	1.39
C.40	C.40	390	C.40	1.40	1.40
C.41	C.41	400	C.41	1.41	1.41
C.42	C.42	410	C.42	1.42	1.42
C.43	C.43	420	C.43	1.43	1.43
C.44	C.44	430	C.44	1.44	1.44
C.45	C.45	440	C.45	1.45	1.45
C.46	C.46	450	C.46	1.46	1.46
C.47	C.47	460	C.47	1.47	1.47
C.48	C.48	470	C.48	1.48	1.48
C.49	C.49	480	C.49	1.49	1.49
C.50	C.50	490	C.50	1.50	1.50
C.51	C.51	500	C.51	1.51	1.51
C.52	C.52	510	C.52	1.52	1.52
C.53	C.53	520	C.53	1.53	1.53
C.54	C.54	530	C.54	1.54	1.54
C.55	C.55	540	C.55	1.55	1.55
C.56	C.56	550	C.56	1.56	1.56
C.57	C.57	560	C.57	1.57	1.57
C.58	C.58	570	C.58	1.58	1.58
C.59	C.59	580	C.59	1.59	1.59
C.60	C.60	590	C.60	1.60	1.60
C.61	C.61	600	C.61	1.61	1.61
C.62	C.62	610	C.62	1.62	1.62
C.63	C.63	620	C.63	1.63	1.63
C.64	C.64	630	C.64	1.64	1.64
C.65	C.65	640	C.65	1.65	1.65
C.66	C.66	650	C.66	1.66	1.66
C.67	C.67	660	C.67	1.67	1.67
C.68	C.68	670	C.68	1.68	1.68
C.69	C.69	680	C.69	1.69	1.69
C.70	C.70	690	C.70	1.70	1.70
C.71	C.71	700	C.71	1.71	1.71
C.72	C.72	710	C.72	1.72	1.72
C.73	C.73	720	C.73	1.73	1.73
C.74	C.74	730	C.74	1.74	1.74
C.75	C.75	740	C.75	1.75	1.75
C.76	C.76	750	C.76	1.76	1.76
C.77	C.77	760	C.77	1.77	1.77
C.78	C.78	770	C.78	1.78	1.78
C.79	C.79	780	C.79	1.79	1.79
C.80	C.80	790	C.80	1.80	1.80
C.81	C.81	800	C.81	1.81	1.81
C.82	C.82	810	C.82	1.82	1.82
C.83	C.83	820	C.83	1.83	1.83
C.84	C.84	830	C.84	1.84	1.84
C.85	C.85	840	C.85	1.85	1.85
C.86	C.86	850	C.86	1.86	1.86
C.87	C.87	860	C.87	1.87	1.87
C.88	C.88	870	C.88	1.88	1.88
C.89	C.89	880	C.89	1.89	1.89
C.90	C.90	890	C.90	1.90	1.90
C.91	C.91	900	C.91	1.91	1.91
C.92	C.92	910	C.92	1.92	1.92
C.93	C.93	920	C.93	1.93	1.93
C.94	C.94	930	C.94	1.94	1.94
C.95	C.95	940	C.95	1.95	1.95
C.96	C.96	950	C.96	1.96	1.96
C.97	C.97	960	C.97	1.97	1.97
C.98	C.98	970	C.98	1.98	1.98
C.99	C.99	980	C.99	1.99	1.99
C.100	C.100	990	C.100	2.00	2.00

TABLE I (Continued)

hk $\ell$	Calc.	Obs.	hk $\ell$	Calc.	Obs.
501	- 8.3	9.5	<u>661</u>	+14.2	11.8
511	- 1.5	0	<u>671</u>	- 7.2	0
521	- 6.8	4.6	<u>681</u>	+ 4.0	0
531	-11.5	12.3	<u>691</u>	+ 2.1	0
541	- 6.0	6.6	<u>6,10,1</u>	- 2.0	0
551	+ 4.4	5.2	<u>6,11,1</u>	- 1.9	0
561	- 8.2	8.0			
571	+ 6.2	0	701	+ 5.0	0
581	- 3.7	0	711	+ 3.2	0
591	+ 9.1	7.5	721	+ 4.9	0
5,10,1	+ 0.5	0	731	+ 5.6	0
5,11,1	+ 1.0	0	741	+ 4.4	4.3
			751	- 0.8	0
<u>501</u>	-14.4	19.2	761	+ 6.2	6.3
<u>511</u>	-11.8	15.5	771	- 4.8	0
<u>521</u>	+21.1	27.0	781	+ 2.6	0
<u>531</u>	+22.8	23.0	791	- 4.4	0
<u>541</u>	-20.0	20.6			
<u>551</u>	-14.9	14.1	<u>701</u>	-15.0	21.5
<u>561</u>	+14.6	14.6	<u>711</u>	-10.1	11.8
<u>571</u>	- 2.6	0	<u>721</u>	+11.2	11.2
<u>581</u>	-10.0	6.6	<u>731</u>	+ 2.1	0
<u>591</u>	-18.2	14.6	<u>741</u>	- 6.4	8.6
<u>5,10,1</u>	+ 8.0	0	<u>751</u>	- 4.3	5.7
<u>5,11,1</u>	+ 1.4	0	<u>761</u>	+ 9.5	9.8
<u>5,12,1</u>	- 5.3	0	<u>771</u>	+12.6	9.8
			<u>781</u>	- 6.6	7.8
601	-19.8	24.6	<u>791</u>	+ 3.1	2.6
611	+ 5.4	5.2	<u>7,10,1</u>	+ 5.0	0
621	+16.5	22.6			
631	- 3.2	5.5	801	- 0.6	0
641	- 4.0	6.3	811	- 1.2	0
651	+ 4.7	4.6	821	+ 1.6	4.6
661	+19.8	19.2	831	+ 0.5	3.4
671	+ 2.1	0	841	- 8.6	8.6
681	- 6.4	7.2	851	- 1.6	0
691	+ 7.7	7.5	861	- 1.7	2.9
6,10,1	+ 4.5	4.0	871	- 2.8	0
<u>601</u>	+ 0.1	0	<u>801</u>	- 1.8	0
<u>611</u>	+ 7.3	7.7	<u>811</u>	+ 1.0	0
<u>621</u>	+ 6.6	5.5	<u>821</u>	- 2.7	0
<u>631</u>	- 0.7	4.0	<u>831</u>	- 5.2	5.2
<u>641</u>	+12.9	11.2	<u>841</u>	- 8.8	6.9
<u>651</u>	+ 2.0	0	<u>851</u>	+ 3.6	3.4



Year	Value	Year	Value	Year	Value
1911	1.11	1912	1.12	1913	1.13
1914	1.14	1915	1.15	1916	1.16
1917	1.17	1918	1.18	1919	1.19
1920	1.20	1921	1.21	1922	1.22
1923	1.23	1924	1.24	1925	1.25
1926	1.26	1927	1.27	1928	1.28
1929	1.29	1930	1.30	1931	1.31
1932	1.32	1933	1.33	1934	1.34
1935	1.35	1936	1.36	1937	1.37
1938	1.38	1939	1.39	1940	1.40
1941	1.41	1942	1.42	1943	1.43
1944	1.44	1945	1.45	1946	1.46
1947	1.47	1948	1.48	1949	1.49
1950	1.50	1951	1.51	1952	1.52
1953	1.53	1954	1.54	1955	1.55
1956	1.56	1957	1.57	1958	1.58
1959	1.59	1960	1.60	1961	1.61
1962	1.62	1963	1.63	1964	1.64
1965	1.65	1966	1.66	1967	1.67
1968	1.68	1969	1.69	1970	1.70
1971	1.71	1972	1.72	1973	1.73
1974	1.74	1975	1.75	1976	1.76
1977	1.77	1978	1.78	1979	1.79
1980	1.80	1981	1.81	1982	1.82
1983	1.83	1984	1.84	1985	1.85
1986	1.86	1987	1.87	1988	1.88
1989	1.89	1990	1.90	1991	1.91
1992	1.92	1993	1.93	1994	1.94
1995	1.95	1996	1.96	1997	1.97
1998	1.98	1999	1.99	2000	2.00



TABLE I (Continued)

hk $\ell$	Calc.	Obs.	hk $\ell$	Calc.	Obs.
<u>861</u>	- 7.7	5.2	192	+11.7	8.3
<u>871</u>	- 0.5	0	1,10,2	- 6.2	0
<u>881</u>	- 3.5	3.1	1,11,2	- 7.8	6.3
901	- 4.1	0	1,12,2	+ 7.0	5.5
911	- 2.3	0	1,13,2	- 2.9	3.4
921	- 1.6	0	<u>102</u>	+12.5	16.1
931	- 3.0	0	<u>112</u>	+20.9	21.0
941	- 4.0	0	<u>122</u>	-44.5	44.8
<u>901</u>	+ 0.5	0	<u>132</u>	+10.3	13.8
<u>911</u>	- 1.2	0	<u>142</u>	+12.0	12.1
<u>921</u>	- 0.3	0	<u>152</u>	+21.6	18.1
<u>931</u>	+ 5.1	4.0	<u>162</u>	-25.7	21.0
<u>941</u>	- 3.9	5.4	<u>172</u>	-10.1	7.5
<u>951</u>	- 3.0	0	<u>182</u>	+ 9.0	0
<u>961</u>	- 0.6	0	<u>192</u>	- 0.2	0
<u>10,0,1</u>	+ 2.2	0	1,10,2	-11.5	10.6
<u>10,1,1</u>	+ 1.5	0	1,11,2	+ 1.1	0
<u>10,2,1</u>	+ 1.0	0	1,12,2	+ 6.1	0
002	+32.6	38.4	1,13,2	+ 9.1	7.5
012	+ 0.1	0	202	-14.6	13.5
022	+18.1	21.2	212	+ 5.4	4.9
032	+ 9.4	10.1	222	-19.5	21.8
042	+ 4.0	5.2	232	-14.3	15.5
052	+ 4.7	2.9	242	+ 5.5	4.0
062	-11.6	11.8	252	- 1.0	0
072	+ 8.0	5.2	262	+ 4.9	4.9
082	+ 2.3	0	272	-10.2	3.4
092	+ 4.2	3.4	282	+ 3.0	0
0,10,2	+ 7.5	7.2	292	- 1.1	0
0,11,2	+ 5.4	5.7	2,10,2	- 9.4	7.8
0,12,2	+ 5.4	4.3	2,11,2	- 6.9	4.6
0,13,2	+ 2.8	4.0	2,12,2	- 2.6	0
102	+46.0	46.5	<u>202</u>	-28.2	33.0
112	+ 8.1	9.8	<u>212</u>	-22.0	22.4
122	- 8.2	9.2	<u>222</u>	-24.9	28.4
132	-32.2	31.8	<u>232</u>	+12.6	11.8
142	+32.7	31.0	<u>242</u>	- 1.3	0
152	+ 0.4	0	<u>252</u>	-18.4	12.9
162	- 9.5	7.2	<u>262</u>	+ 6.2	5.2
172	- 4.1	0	<u>272</u>	+ 3.9	2.3
182	+16.5	15.2	<u>282</u>	- 0.2	0
			<u>292</u>	-12.9	9.5
			2,10,2	- 9.8	8.6

TABLE 2 (Continued)

Year	Age	Sex	Year	Age	Sex
1950	7.11	Male	1950	7.7	Male
1951	7.11	Male	1951	7.7	Male
1952	7.11	Male	1952	7.7	Male
1953	7.11	Male	1953	7.7	Male
1954	7.11	Male	1954	7.7	Male
1955	7.11	Male	1955	7.7	Male
1956	7.11	Male	1956	7.7	Male
1957	7.11	Male	1957	7.7	Male
1958	7.11	Male	1958	7.7	Male
1959	7.11	Male	1959	7.7	Male
1960	7.11	Male	1960	7.7	Male
1961	7.11	Male	1961	7.7	Male
1962	7.11	Male	1962	7.7	Male
1963	7.11	Male	1963	7.7	Male
1964	7.11	Male	1964	7.7	Male
1965	7.11	Male	1965	7.7	Male
1966	7.11	Male	1966	7.7	Male
1967	7.11	Male	1967	7.7	Male
1968	7.11	Male	1968	7.7	Male
1969	7.11	Male	1969	7.7	Male
1970	7.11	Male	1970	7.7	Male
1971	7.11	Male	1971	7.7	Male
1972	7.11	Male	1972	7.7	Male
1973	7.11	Male	1973	7.7	Male
1974	7.11	Male	1974	7.7	Male
1975	7.11	Male	1975	7.7	Male
1976	7.11	Male	1976	7.7	Male
1977	7.11	Male	1977	7.7	Male
1978	7.11	Male	1978	7.7	Male
1979	7.11	Male	1979	7.7	Male
1980	7.11	Male	1980	7.7	Male
1981	7.11	Male	1981	7.7	Male
1982	7.11	Male	1982	7.7	Male
1983	7.11	Male	1983	7.7	Male
1984	7.11	Male	1984	7.7	Male
1985	7.11	Male	1985	7.7	Male
1986	7.11	Male	1986	7.7	Male
1987	7.11	Male	1987	7.7	Male
1988	7.11	Male	1988	7.7	Male
1989	7.11	Male	1989	7.7	Male
1990	7.11	Male	1990	7.7	Male
1991	7.11	Male	1991	7.7	Male
1992	7.11	Male	1992	7.7	Male
1993	7.11	Male	1993	7.7	Male
1994	7.11	Male	1994	7.7	Male
1995	7.11	Male	1995	7.7	Male
1996	7.11	Male	1996	7.7	Male
1997	7.11	Male	1997	7.7	Male
1998	7.11	Male	1998	7.7	Male
1999	7.11	Male	1999	7.7	Male
2000	7.11	Male	2000	7.7	Male

TABLE 1 (Continued)

hk $\ell$	Calc.	Obs.	hk $\ell$	Calc.	Obs.
$\bar{2},11,2$	+ 1.4	0	$\bar{4}02$	+22.2	26.1
$\bar{2},12,2$	- 4.0	3.7	$\bar{4}12$	-13.4	14.6
$\bar{2},13,2$	- 6.0	5.2	$\bar{4}22$	+21.8	19.5
302	- 9.1	7.5	$\bar{4}32$	+14.2	14.1
312	+21.1	20.1	$\bar{4}42$	+ 0.9	3.1
322	-19.9	22.4	$\bar{4}52$	- 7.8	9.8
332	- 1.3	0	$\bar{4}62$	- 3.2	4.9
342	- 3.2	0	$\bar{4}72$	+13.0	10.0
352	+19.4	16.1	$\bar{4}82$	0	0
362	- 9.4	7.5	$\bar{4}92$	- 1.4	0
372	-14.0	7.8	$\bar{4},10,2$	+ 8.8	8.3
382	- 0.3	0	$\bar{4},11,2$	+ 7.0	5.7
392	- 2.0	0	$\bar{4},12,2$	+ 3.2	3.7
$3,10,2$	- 5.3	4.0	502	+ 6.0	0
$3,11,2$	- 3.0	0	512	- 3.9	4.9
$3,12,2$	+ 0.6	0	522	+15.6	16.9
$\bar{3}02$	+27.0	36.0	532	- 7.7	8.6
$\bar{3}12$	+ 2.8	4.6	542	+ 1.3	0
$\bar{3}22$	- 3.5	5.2	552	- 4.9	0
$\bar{3}32$	-28.8	29.3	562	+ 7.1	6.9
$\bar{3}42$	+20.8	21.0	572	+ 4.1	0
$\bar{3}52$	- 4.3	6.0	582	- 0.4	0
$\bar{3}62$	- 2.4	4.0	592	+ 4.1	0
$\bar{3}72$	- 3.6	0	$5,10,2$	+ 5.0	5.2
$\bar{3}82$	+10.7	10.9	$\bar{5}02$	- 3.8	2.3
$\bar{3}92$	+ 7.8	5.7	$\bar{5}12$	+17.6	18.4
$\bar{3},10,2$	- 4.2	0	$\bar{5}22$	- 7.6	8.6
$\bar{3},11,2$	- 7.6	7.2	$\bar{5}32$	+ 3.9	3.7
$\bar{3},12,2$	+ 3.7	0	$\bar{5}42$	- 3.9	0
$3,13,2$	- 4.8	0	$\bar{5}52$	+18.3	20.1
402	+20.2	20.4	$\bar{5}62$	- 6.8	8.9
412	+10.3	8.3	$\bar{5}72$	- 8.2	6.9
422	+ 0.3	0	$\bar{5}82$	- 1.4	0
432	- 1.5	0	$\bar{5}92$	+ 2.8	0
442	+ 5.0	5.7	$\bar{5},10,2$	- 0.8	0
452	+12.5	8.6	$\bar{5},11,2$	+ 0.4	0
462	-13.1	10.4	$\bar{5},12,2$	+ 0.8	0
472	- 1.3	0	602	+ 1.3	6.0
482	+ 3.7	0	612	- 2.3	0
492	+ 7.1	0	622	-11.6	12.6
$4,10,2$	+ 1.7	0	632	- 4.0	2.9
$4,11,2$	+ 1.0	0	642	+ 9.2	5.7



TABLE 1. Continued

Year	Value	Unit	Year	Value	Unit
1971	2.35	kg	1971	0.1	kg
1972	2.15	kg	1972	0.1	kg
1973	2.15	kg	1973	0.1	kg
1974	2.15	kg	1974	0.1	kg
1975	2.15	kg	1975	0.1	kg
1976	2.15	kg	1976	0.1	kg
1977	2.15	kg	1977	0.1	kg
1978	2.15	kg	1978	0.1	kg
1979	2.15	kg	1979	0.1	kg
1980	2.15	kg	1980	0.1	kg
1981	2.15	kg	1981	0.1	kg
1982	2.15	kg	1982	0.1	kg
1983	2.15	kg	1983	0.1	kg
1984	2.15	kg	1984	0.1	kg
1985	2.15	kg	1985	0.1	kg
1986	2.15	kg	1986	0.1	kg
1987	2.15	kg	1987	0.1	kg
1988	2.15	kg	1988	0.1	kg
1989	2.15	kg	1989	0.1	kg
1990	2.15	kg	1990	0.1	kg
1991	2.15	kg	1991	0.1	kg
1992	2.15	kg	1992	0.1	kg
1993	2.15	kg	1993	0.1	kg
1994	2.15	kg	1994	0.1	kg
1995	2.15	kg	1995	0.1	kg
1996	2.15	kg	1996	0.1	kg
1997	2.15	kg	1997	0.1	kg
1998	2.15	kg	1998	0.1	kg
1999	2.15	kg	1999	0.1	kg
2000	2.15	kg	2000	0.1	kg
2001	2.15	kg	2001	0.1	kg
2002	2.15	kg	2002	0.1	kg
2003	2.15	kg	2003	0.1	kg
2004	2.15	kg	2004	0.1	kg
2005	2.15	kg	2005	0.1	kg
2006	2.15	kg	2006	0.1	kg
2007	2.15	kg	2007	0.1	kg
2008	2.15	kg	2008	0.1	kg
2009	2.15	kg	2009	0.1	kg
2010	2.15	kg	2010	0.1	kg
2011	2.15	kg	2011	0.1	kg
2012	2.15	kg	2012	0.1	kg
2013	2.15	kg	2013	0.1	kg
2014	2.15	kg	2014	0.1	kg
2015	2.15	kg	2015	0.1	kg
2016	2.15	kg	2016	0.1	kg
2017	2.15	kg	2017	0.1	kg
2018	2.15	kg	2018	0.1	kg
2019	2.15	kg	2019	0.1	kg
2020	2.15	kg	2020	0.1	kg



TABLE I (Continued)

hk l	Calc.	Obs.	hk l	Calc.	Obs.
652	- 4.8	5.2	802	+ 9.7	9.5
662	- 0.3	0	812	+ 9.7	11.8
672	- 2.0	0	822	+11.0	8.6
682	+ 5.6	5.5	832	-10.3	10.6
692	- 2.2	0	842	+ 0.7	0
702	-22.8	21.8	852	+ 8.0	8.6
712	+ 2.2	0	862	- 0.5	0
722	- 8.9	9.2	872	- 4.6	5.2
732	- 2.0	4.3	882	- 0.1	0
742	- 7.5	6.6	902	- 1.2	0
752	+ 0.7	0	912	+ 4.1	0
762	+ 6.6	5.2	922	+ 0.7	0
772	- 4.9	4.0	932	+ 4.3	0
782	- 4.2	0	942	- 2.4	0
792	- 2.2	0	952	+ 5.7	4.3
6,10,2	- 4.1	4.9	962	- 1.3	0
6,11,2	- 2.3	0	10,3,2	- 6.4	0
702	-13.2	14.3	10,1,2	+ 7.5	0
712	- 3.2	4.6	10,2,2	- 5.8	0
722	- 1.2	0	10,3,2	- 5.8	0
732	+ 9.1	7.8	003	+25.4	31.6
742	- 8.3	8.0	013	+11.9	13.2
752	- 1.8	0	023	- 5.4	4.6
762	+ 2.6	3.7	033	-10.9	9.5
772	+ 0.7	0	043	- 2.2	2.0
702	+ 0.1	0	053	- 2.1	0
712	+ 3.0	5.7	063	-18.5	16.4
722	+ 3.9	4.6	073	-13.1	10.6
732	-16.1	16.1	083	+ 3.5	0
742	+ 1.5	0	093	- 1.1	0
752	- 1.4	0	0,10,3	+ 1.0	0
762	+ 5.1	5.7	0,11,3	- 8.1	7.8
772	- 4.0	4.6	0,12,3	+ 5.8	7.5
782	+ 0.4	0	103	-18.0	18.4
792	+ 4.7	5.4	113	-19.7	18.7
7,10,2	+ 0.2	0	123	- 5.8	5.2
802	+ 8.2	8.3	133	- 2.7	3.4
812	- 1.9	0	143	-21.7	24.1
822	- 3.5	4.6	153	-11.4	7.2
832	+ 5.9	6.6	163	- 1.7	0
842	+ 2.8	4.3	173	+14.1	8.9
852	- 0.3	0			

TABLE 2 (Continued)

Year	Value	Unit	Year	Value	Unit
1950	1.0	100	1950	1.0	100
1951	1.1	100	1951	1.1	100
1952	1.2	100	1952	1.2	100
1953	1.3	100	1953	1.3	100
1954	1.4	100	1954	1.4	100
1955	1.5	100	1955	1.5	100
1956	1.6	100	1956	1.6	100
1957	1.7	100	1957	1.7	100
1958	1.8	100	1958	1.8	100
1959	1.9	100	1959	1.9	100
1960	2.0	100	1960	2.0	100
1961	2.1	100	1961	2.1	100
1962	2.2	100	1962	2.2	100
1963	2.3	100	1963	2.3	100
1964	2.4	100	1964	2.4	100
1965	2.5	100	1965	2.5	100
1966	2.6	100	1966	2.6	100
1967	2.7	100	1967	2.7	100
1968	2.8	100	1968	2.8	100
1969	2.9	100	1969	2.9	100
1970	3.0	100	1970	3.0	100
1971	3.1	100	1971	3.1	100
1972	3.2	100	1972	3.2	100
1973	3.3	100	1973	3.3	100
1974	3.4	100	1974	3.4	100
1975	3.5	100	1975	3.5	100
1976	3.6	100	1976	3.6	100
1977	3.7	100	1977	3.7	100
1978	3.8	100	1978	3.8	100
1979	3.9	100	1979	3.9	100
1980	4.0	100	1980	4.0	100
1981	4.1	100	1981	4.1	100
1982	4.2	100	1982	4.2	100
1983	4.3	100	1983	4.3	100
1984	4.4	100	1984	4.4	100
1985	4.5	100	1985	4.5	100
1986	4.6	100	1986	4.6	100
1987	4.7	100	1987	4.7	100
1988	4.8	100	1988	4.8	100
1989	4.9	100	1989	4.9	100
1990	5.0	100	1990	5.0	100
1991	5.1	100	1991	5.1	100
1992	5.2	100	1992	5.2	100
1993	5.3	100	1993	5.3	100
1994	5.4	100	1994	5.4	100
1995	5.5	100	1995	5.5	100
1996	5.6	100	1996	5.6	100
1997	5.7	100	1997	5.7	100
1998	5.8	100	1998	5.8	100
1999	5.9	100	1999	5.9	100
2000	6.0	100	2000	6.0	100
2001	6.1	100	2001	6.1	100
2002	6.2	100	2002	6.2	100
2003	6.3	100	2003	6.3	100
2004	6.4	100	2004	6.4	100
2005	6.5	100	2005	6.5	100
2006	6.6	100	2006	6.6	100
2007	6.7	100	2007	6.7	100
2008	6.8	100	2008	6.8	100
2009	6.9	100	2009	6.9	100
2010	7.0	100	2010	7.0	100
2011	7.1	100	2011	7.1	100
2012	7.2	100	2012	7.2	100
2013	7.3	100	2013	7.3	100
2014	7.4	100	2014	7.4	100
2015	7.5	100	2015	7.5	100
2016	7.6	100	2016	7.6	100
2017	7.7	100	2017	7.7	100
2018	7.8	100	2018	7.8	100
2019	7.9	100	2019	7.9	100
2020	8.0	100	2020	8.0	100

TABLE I (Continued)

hk $\ell$	Calc.	Obs.	hk $\ell$	Calc.	Obs.
183	- 8.0	0	303	- 4.7	7.5
193	+ 1.1	0	313	+ 1.8	0
1,10,3	+ 0.4	0	323	+20.7	22.1
1,11,3	+ 3.8	0	333	+15.7	12.1
1,12,3	- 2.4	0	343	+ 2.3	4.6
$\bar{1}$ 03	+ 7.0	0	353	+ 2.2	0
$\bar{1}$ 13	- 5.6	6.0	363	+14.6	12.3
$\bar{1}$ 23	+19.8	24.4	373	- 1.4	0
$\bar{1}$ 33	+28.8	26.4	383	- 3.0	0
$\bar{1}$ 43	+11.6	11.8	393	- 6.0	0
$\bar{1}$ 53	-10.6	7.5	3,10,3	+ 6.1	0
$\bar{1}$ 63	+ 9.8	10.3	3,11,3	+ 2.0	0
$\bar{1}$ 73	+ 0.5	0	$\bar{3}$ 03	-14.8	14.9
$\bar{1}$ 83	+ 1.4	0	$\bar{3}$ 13	-23.2	21.2
$\bar{1}$ 93	-14.3	11.8	$\bar{3}$ 23	-11.5	10.3
$\bar{1}$ ,10,3	+ 4.9	4.6	$\bar{3}$ 33	+ 0.8	0
$\bar{1}$ ,11,3	+ 0.2	0	$\bar{3}$ 43	-15.5	15.2
$\bar{1}$ ,12,3	- 0.6	0	$\bar{3}$ 53	-11.1	7.8
203	- 7.3	4.6	$\bar{3}$ 63	- 3.2	3.7
213	+13.7	14.9	$\bar{3}$ 73	+18.4	16.4
223	-11.8	12.3	$\bar{3}$ 83	- 4.8	0
233	-13.4	16.1	$\bar{3}$ 93	+ 1.6	0
243	+11.7	12.0	$\bar{3}$ ,10,3	- 2.2	0
253	+19.5	17.8	$\bar{3}$ ,11,3	+ 6.2	6.0
263	+ 4.1	0	$\bar{3}$ ,12,3	- 1.6	0
273	- 2.6	0	403	+ 8.0	4.6
283	+ 4.3	0	413	+ 8.8	13.2
293	+12.0	8.3	423	+ 8.4	7.8
2,10,3	- 6.6	0	433	- 6.4	6.9
2,11,3	- 0.5	0	443	- 5.9	5.2
2,12,3	- 1.2	0	453	+ 0.3	0
$\bar{2}$ 03	-13.2	20.4	463	- 3.4	0
$\bar{2}$ 13	-11.5	12.0	473	- 9.9	8.0
$\bar{2}$ 23	- 1.9	0	483	- 1.8	0
$\bar{2}$ 33	+12.2	10.1	493	- 0.6	0
$\bar{2}$ 43	+12.7	10.1	4,10,3	+ 4.8	2.9
$\bar{2}$ 53	+ 2.2	0	$\bar{4}$ 03	+ 0.5	0
$\bar{2}$ 63	+12.5	10.1	$\bar{4}$ 13	-17.5	15.2
$\bar{2}$ 73	+12.4	9.8	$\bar{4}$ 23	+ 7.6	8.6
$\bar{2}$ 83	+ 2.2	0	$\bar{4}$ 33	+12.8	11.4
$\bar{2}$ 93	+ 0.3	0	$\bar{4}$ 43	-19.5	16.6
$\bar{2}$ ,10,3	- 4.2	0	$\bar{4}$ 53	-21.4	17.5
$\bar{2}$ ,11,3	+ 7.9	7.2	$\bar{4}$ 63	+ 6.2	0
$\bar{2}$ ,12,3	- 3.3	4.3	$\bar{4}$ 73	+ 6.5	0



TABLE 1 (Continued)

Year	Value	Unit	Year	Value	Unit
1970	7.4	kg	1970	0.8	kg
1971	10.1	kg	1971	1.4	kg
1972	7.05	kg	1972	0.8	kg
1973	7.05	kg	1973	0.8	kg
1974	1.5	kg	1974	0.8	kg
1975	0.8	kg	1975	0.8	kg
1976	0.8	kg	1976	0.8	kg
1977	0.8	kg	1977	0.8	kg
1978	0.8	kg	1978	0.8	kg
1979	0.8	kg	1979	0.8	kg
1980	0.8	kg	1980	0.8	kg
1981	0.8	kg	1981	0.8	kg
1982	0.8	kg	1982	0.8	kg
1983	0.8	kg	1983	0.8	kg
1984	0.8	kg	1984	0.8	kg
1985	0.8	kg	1985	0.8	kg
1986	0.8	kg	1986	0.8	kg
1987	0.8	kg	1987	0.8	kg
1988	0.8	kg	1988	0.8	kg
1989	0.8	kg	1989	0.8	kg
1990	0.8	kg	1990	0.8	kg
1991	0.8	kg	1991	0.8	kg
1992	0.8	kg	1992	0.8	kg
1993	0.8	kg	1993	0.8	kg
1994	0.8	kg	1994	0.8	kg
1995	0.8	kg	1995	0.8	kg
1996	0.8	kg	1996	0.8	kg
1997	0.8	kg	1997	0.8	kg
1998	0.8	kg	1998	0.8	kg
1999	0.8	kg	1999	0.8	kg
2000	0.8	kg	2000	0.8	kg
2001	0.8	kg	2001	0.8	kg
2002	0.8	kg	2002	0.8	kg
2003	0.8	kg	2003	0.8	kg
2004	0.8	kg	2004	0.8	kg
2005	0.8	kg	2005	0.8	kg
2006	0.8	kg	2006	0.8	kg
2007	0.8	kg	2007	0.8	kg
2008	0.8	kg	2008	0.8	kg
2009	0.8	kg	2009	0.8	kg
2010	0.8	kg	2010	0.8	kg
2011	0.8	kg	2011	0.8	kg
2012	0.8	kg	2012	0.8	kg
2013	0.8	kg	2013	0.8	kg
2014	0.8	kg	2014	0.8	kg
2015	0.8	kg	2015	0.8	kg
2016	0.8	kg	2016	0.8	kg
2017	0.8	kg	2017	0.8	kg
2018	0.8	kg	2018	0.8	kg
2019	0.8	kg	2019	0.8	kg
2020	0.8	kg	2020	0.8	kg



TABLE I (Continued)

hk $\ell$	Calc.	Obs.	hk $\ell$	Calc.	Obs.
$\overline{483}$	- 6.9	6.9	$\overline{653}$	- 4.6	3.2
$\overline{493}$	-11.5	7.8	$\overline{663}$	+12.5	11.8
$\overline{4,10,3}$	+ 6.4	6.3	$\overline{673}$	+11.2	10.1
$\overline{4,11,3}$	+ 1.5	0	$\overline{683}$	- 0.1	0
$\overline{4,12,3}$	+ 0.6	0	$\overline{693}$	- 4.6	0
503	-13.1	11.4	$\overline{6,10,3}$	- 0.5	0
513	- 5.6	0	703	- 3.2	0
523	+ 3.0	0	713	+ 1.8	3.7
533	- 5.7	0	723	+ 7.7	6.9
543	-17.0	16.6	733	+ 4.8	2.9
553	- 4.2	0	743	+ 1.3	2.3
563	+ 2.2	0	753	+ 1.8	0
573	+ 3.6	0	763	+ 6.4	0
583	- 7.3	0	$\overline{703}$	-10.4	10.6
593	+ 2.6	0	$\overline{713}$	+ 0.4	0
$\overline{503}$	+ 4.6	5.2	$\overline{723}$	- 3.3	6.0
$\overline{513}$	+ 4.5	4.3	$\overline{733}$	-10.3	9.2
$\overline{523}$	+15.2	12.4	$\overline{743}$	- 8.7	8.3
$\overline{533}$	+13.2	12.6	$\overline{753}$	+ 5.0	3.7
$\overline{543}$	+ 5.1	6.3	$\overline{763}$	+ 1.5	0
$\overline{553}$	- 4.3	0	$\overline{773}$	+ 3.5	0
$\overline{563}$	+ 6.4	0	$\overline{783}$	- 3.7	0
$\overline{573}$	- 5.7	0	$\overline{793}$	+ 7.8	6.0
$\overline{583}$	- 0.2	0	803	+ 1.1	0
$\overline{593}$	- 9.1	10.4	813	- 0.3	0
$\overline{5,10,3}$	+ 4.8	0	$\overline{803}$	+ 4.3	5.7
$\overline{5,11,3}$	- 0.5	0	$\overline{813}$	- 6.8	10.3
603	-13.0	11.2	$\overline{823}$	- 3.6	0
613	- 2.5	0	$\overline{833}$	+ 3.4	6.3
623	+ 1.0	0	$\overline{843}$	- 6.1	0
633	+ 0.2	0	$\overline{853}$	- 8.8	10.9
643	- 3.6	6.6	$\overline{863}$	- 8.0	7.2
653	+ 2.4	0	$\overline{873}$	+ 2.8	5.7
663	+ 7.1	7.2	$\overline{883}$	- 0.7	0
673	+ 4.5	0	$\overline{903}$	+ 3.1	0
683	- 3.3	0	$\overline{913}$	+ 8.2	9.5
$\overline{603}$	- 9.2	12.4	$\overline{923}$	+ 4.6	5.5
$\overline{613}$	-12.6	14.9	$\overline{933}$	- 1.9	0
$\overline{623}$	+ 5.8	6.0	$\overline{943}$	+ 1.9	0
$\overline{633}$	+15.6	14.6	$\overline{953}$	+ 3.5	4.6
$\overline{643}$	+ 7.0	4.9	$\overline{963}$	+ 0.8	0

TABLE 1 (Continued)

Year	Calc.	Obs.	Year	Calc.	Obs.
1950	1.0	1.0	1955	1.0	1.0
1951	1.1	1.1	1956	1.1	1.1
1952	1.2	1.2	1957	1.2	1.2
1953	1.3	1.3	1958	1.3	1.3
1954	1.4	1.4	1959	1.4	1.4
1960	1.5	1.5	1960	1.5	1.5
1961	1.6	1.6	1961	1.6	1.6
1962	1.7	1.7	1962	1.7	1.7
1963	1.8	1.8	1963	1.8	1.8
1964	1.9	1.9	1964	1.9	1.9
1965	2.0	2.0	1965	2.0	2.0
1966	2.1	2.1	1966	2.1	2.1
1967	2.2	2.2	1967	2.2	2.2
1968	2.3	2.3	1968	2.3	2.3
1969	2.4	2.4	1969	2.4	2.4
1970	2.5	2.5	1970	2.5	2.5
1971	2.6	2.6	1971	2.6	2.6
1972	2.7	2.7	1972	2.7	2.7
1973	2.8	2.8	1973	2.8	2.8
1974	2.9	2.9	1974	2.9	2.9
1975	3.0	3.0	1975	3.0	3.0
1976	3.1	3.1	1976	3.1	3.1
1977	3.2	3.2	1977	3.2	3.2
1978	3.3	3.3	1978	3.3	3.3
1979	3.4	3.4	1979	3.4	3.4
1980	3.5	3.5	1980	3.5	3.5
1981	3.6	3.6	1981	3.6	3.6
1982	3.7	3.7	1982	3.7	3.7
1983	3.8	3.8	1983	3.8	3.8
1984	3.9	3.9	1984	3.9	3.9
1985	4.0	4.0	1985	4.0	4.0
1986	4.1	4.1	1986	4.1	4.1
1987	4.2	4.2	1987	4.2	4.2
1988	4.3	4.3	1988	4.3	4.3
1989	4.4	4.4	1989	4.4	4.4
1990	4.5	4.5	1990	4.5	4.5
1991	4.6	4.6	1991	4.6	4.6
1992	4.7	4.7	1992	4.7	4.7
1993	4.8	4.8	1993	4.8	4.8
1994	4.9	4.9	1994	4.9	4.9
1995	5.0	5.0	1995	5.0	5.0
1996	5.1	5.1	1996	5.1	5.1
1997	5.2	5.2	1997	5.2	5.2
1998	5.3	5.3	1998	5.3	5.3
1999	5.4	5.4	1999	5.4	5.4
2000	5.5	5.5	2000	5.5	5.5
2001	5.6	5.6	2001	5.6	5.6
2002	5.7	5.7	2002	5.7	5.7
2003	5.8	5.8	2003	5.8	5.8
2004	5.9	5.9	2004	5.9	5.9
2005	6.0	6.0	2005	6.0	6.0
2006	6.1	6.1	2006	6.1	6.1
2007	6.2	6.2	2007	6.2	6.2
2008	6.3	6.3	2008	6.3	6.3
2009	6.4	6.4	2009	6.4	6.4
2010	6.5	6.5	2010	6.5	6.5
2011	6.6	6.6	2011	6.6	6.6
2012	6.7	6.7	2012	6.7	6.7
2013	6.8	6.8	2013	6.8	6.8
2014	6.9	6.9	2014	6.9	6.9
2015	7.0	7.0	2015	7.0	7.0
2016	7.1	7.1	2016	7.1	7.1
2017	7.2	7.2	2017	7.2	7.2
2018	7.3	7.3	2018	7.3	7.3
2019	7.4	7.4	2019	7.4	7.4
2020	7.5	7.5	2020	7.5	7.5

TABLE I (Continued)

hk $\ell$	Calc.	Obs.	hk $\ell$	Calc.	Obs.
$\overline{10},0,3$	+ 1.9	0	224	- 4.3	4.9
$\overline{10},1,3$	- 2.7	0	234	- 7.0	7.8
$\overline{10},2,3$	- 1.2	0	244	+16.4	14.4
$\overline{10},3,3$	+ 4.5	0	254	- 2.5	0
004	+21.6	28.7	264	+ 4.2	5.2
014	+11.3	10.3	274	- 4.3	0
024	-10.2	8.3	284	+ 4.8	0
034	- 3.3	0	294	- 1.7	0
044	- 3.3	5.5	2,10,4	- 4.1	0
054	+12.7	9.5	$\overline{204}$	- 8.8	13.8
064	-16.8	15.5	$\overline{214}$	+11.8	11.2
074	- 1.1	0	$\overline{224}$	- 6.8	6.6
084	+ 4.4	0	$\overline{234}$	-16.4	16.4
094	+ 7.9	6.6	$\overline{244}$	+15.2	14.6
0,10,4	- 1.8	0	$\overline{254}$	+ 4.5	5.7
0,11,4	+ 0.7	0	$\overline{264}$	+ 5.4	4.9
104	+21.6	32.7	$\overline{274}$	-13.8	11.2
114	+ 1.7	0	$\overline{284}$	+ 3.3	0
124	+ 1.3	0	$\overline{294}$	+ 1.5	0
134	-17.8	18.7	$\overline{2,10,4}$	- 4.6	0
144	+14.9	15.5	$\overline{2,11,4}$	- 8.5	11.8
154	+ 9.1	5.7	304	- 5.8	0
164	- 2.5	4.9	314	+16.5	16.1
174	+ 1.9	0	324	-11.1	13.8
184	+ 8.1	0	334	- 3.2	0
194	+ 9.9	0	344	- 0.9	0
1,10,4	- 1.6	0	354	+ 6.7	6.3
1,11,4	- 0.5	0	364	- 5.2	6.0
$\overline{104}$	- 1.7	0	374	- 13.2	8.9
$\overline{114}$	+ 6.5	7.2	384	+ 0.1	0
$\overline{124}$	-23.3	23.5	394	+ 2.2	0
$\overline{134}$	+13.8	14.9	3,10,4	- 3.4	0
$\overline{144}$	+ 0.6	0	$\overline{304}$	+15.4	19.8
$\overline{154}$	- 2.8	0	$\overline{314}$	-20.7	18.4
$\overline{164}$	+13.5	11.5	$\overline{324}$	+ 8.5	6.9
$\overline{174}$	- 6.2	0	$\overline{334}$	- 1.1	3.4
$\overline{184}$	+ 2.5	0	$\overline{344}$	+12.9	10.6
$\overline{194}$	- 6.4	5.5	$\overline{354}$	- 9.1	8.6
$\overline{1,10,4}$	- 6.7	11.2	$\overline{364}$	+ 3.9	0
$\overline{1,11,4}$	- 0.9	0	$\overline{374}$	+13.8	10.6
204	- 1.2	0	$\overline{384}$	+ 5.5	5.7
214	- 0	0	$\overline{394}$	- 1.0	0
			$\overline{3,10,4}$	+ 0.7	0
			$\overline{3,11,4}$	+ 4.7	0



TABLE 1 (Continued)

Year	Age	Sex	Year	Age	Sex
1948	1.0	M	1948	1.0	M
1949	1.0	M	1949	1.0	M
1950	1.0	M	1950	1.0	M
1951	1.0	M	1951	1.0	M
1952	1.0	M	1952	1.0	M
1953	1.0	M	1953	1.0	M
1954	1.0	M	1954	1.0	M
1955	1.0	M	1955	1.0	M
1956	1.0	M	1956	1.0	M
1957	1.0	M	1957	1.0	M
1958	1.0	M	1958	1.0	M
1959	1.0	M	1959	1.0	M
1960	1.0	M	1960	1.0	M
1961	1.0	M	1961	1.0	M
1962	1.0	M	1962	1.0	M
1963	1.0	M	1963	1.0	M
1964	1.0	M	1964	1.0	M
1965	1.0	M	1965	1.0	M
1966	1.0	M	1966	1.0	M
1967	1.0	M	1967	1.0	M
1968	1.0	M	1968	1.0	M
1969	1.0	M	1969	1.0	M
1970	1.0	M	1970	1.0	M
1971	1.0	M	1971	1.0	M
1972	1.0	M	1972	1.0	M
1973	1.0	M	1973	1.0	M
1974	1.0	M	1974	1.0	M
1975	1.0	M	1975	1.0	M
1976	1.0	M	1976	1.0	M
1977	1.0	M	1977	1.0	M
1978	1.0	M	1978	1.0	M
1979	1.0	M	1979	1.0	M
1980	1.0	M	1980	1.0	M
1981	1.0	M	1981	1.0	M
1982	1.0	M	1982	1.0	M
1983	1.0	M	1983	1.0	M
1984	1.0	M	1984	1.0	M
1985	1.0	M	1985	1.0	M
1986	1.0	M	1986	1.0	M
1987	1.0	M	1987	1.0	M
1988	1.0	M	1988	1.0	M
1989	1.0	M	1989	1.0	M
1990	1.0	M	1990	1.0	M
1991	1.0	M	1991	1.0	M
1992	1.0	M	1992	1.0	M
1993	1.0	M	1993	1.0	M
1994	1.0	M	1994	1.0	M
1995	1.0	M	1995	1.0	M
1996	1.0	M	1996	1.0	M
1997	1.0	M	1997	1.0	M
1998	1.0	M	1998	1.0	M
1999	1.0	M	1999	1.0	M
2000	1.0	M	2000	1.0	M



TABLE I (Continued)

hk $\ell$	Calc.	Obs.	hk $\ell$	Calc.	Obs.
404	+ 2.0	0	604	- 5.4	5.7
414	+ 0.7	0	614	- 4.6	4.6
424	- 0.1	0	624	+ 5.3	5.2
434	+ 5.2	5.2	634	+ 0.2	0
444	-10.6	8.9	644	+ 2.9	2.9
454	+ 1.7	0	654	- 4.1	0
464	- 5.1	0			
474	+ 2.5	0	$\bar{6}04$	-19.8	21.5
484	- 2.5	0	$\bar{6}14$	+ 9.6	10.6
494	+ 1.0	0	$\bar{6}24$	+ 3.4	0
			$\bar{6}34$	-10.0	10.0
$\bar{4}04$	+ 3.7	13.5	$\bar{6}44$	- 1.2	0
$\bar{4}14$	+13.5	14.9	$\bar{6}54$	+ 3.2	0
$\bar{4}24$	+ 8.1	4.9	$\bar{6}64$	+10.2	8.0
$\bar{4}34$	- 9.3	9.8	$\bar{6}74$	-10.7	10.3
$\bar{4}44$	-12.8	10.0	$\bar{6}84$	- 4.6	0
$\bar{4}54$	+15.9	12.4	$\bar{6}94$	+ 8.0	0
$\bar{4}64$	- 3.3	0	$\bar{6},10,4$	+ 0.8	0
$\bar{4}74$	- 2.8	0			
$\bar{4}84$	- 2.9	0	704	- 1.1	0
$\bar{4}94$	+10.7	13.5	714	+ 4.0	0
$\bar{4},10,4$	+ 4.8	0			
$\bar{4},11,4$	- 0.7	0	$\bar{7}04$	+ 2.7	0
			$\bar{7}14$	- 4.1	4.6
504	+ 8.6	0	$\bar{7}24$	+ 9.1	6.3
514	+ 2.3	0	$\bar{7}34$	- 9.0	8.0
524	+ 2.4	0	$\bar{7}44$	+ 4.5	5.2
534	- 9.8	8.0	$\bar{7}54$	- 0.1	0
544	+ 3.8	0	$\bar{7}64$	+ 6.4	0
554	+ 6.6	5.5	$\bar{7}74$	+ 2.1	0
564	- 0.6	0	$\bar{7}84$	+ 0.7	0
574	+ 0.9	0	$\bar{7}94$	+ 2.4	0
$\bar{5}04$	-12.2	11.5	$\bar{8}04$	+ 4.9	0
$\bar{5}14$	- 0.3	0	$\bar{8}14$	+ 4.9	5.4
$\bar{5}24$	- 6.4	6.3	$\bar{8}24$	+ 7.3	8.0
$\bar{5}34$	+17.5	14.6	$\bar{8}34$	- 6.2	6.3
$\bar{5}44$	-12.2	8.0	$\bar{8}44$	- 6.3	9.5
$\bar{5}54$	- 5.4	0	$\bar{8}54$	+ 4.9	6.9
$\bar{5}64$	- 3.1	0	$\bar{8}64$	+ 0.3	0
$\bar{5}74$	+ 0.1	0	$\bar{8}74$	- 2.7	0
$\bar{5}84$	- 5.1	0			
$\bar{5}94$	- 6.8	0	$\bar{9}04$	- 3.2	0
$\bar{5},10,4$	+ 0.1	0	$\bar{9}14$	+ 7.8	10.9



TABLE I (Continued)

hkl	Calc.	Obs.	hkl	Calc.	Obs.
$\overline{9}24$	- 3.4	0	205	-16.0	16.3
$\overline{9}34$	+ 1.1	0	215	- 4.7	0
$\overline{9}44$	- 4.7	0	225	- 6.8	6.0
$\overline{9}54$	+ 4.4	5.7	235	+ 5.5	0
			245	- 2.8	0
$\overline{10},0,4$	- 5.5	6.3	255	- 4.5	3.7
$\overline{10},1,4$	+ 2.3	0	265	+ 3.3	0
$\overline{10},2,4$	- 3.4	0	275		NP
			285		NP
005	+20.9	30.1	295		NP
015	-14.2	12.6			
025	+ 5.5	4.9	$\overline{2}05$	- 7.4	11.2
035	+10.0	9.2	$\overline{2}15$	-13.7	12.6
045	+ 4.5	5.2	$\overline{2}25$	-18.0	14.6
055	-10.7	11.5	$\overline{2}35$	+17.6	13.5
065	- 6.2	5.2	$\overline{2}45$	+ 7.6	4.6
075		NP	$\overline{2}55$	-11.5	0
085		NP	$\overline{2}65$	- 2.3	0
095		NP	$\overline{2}75$		NP
0,10,5		NP	$\overline{2}85$		NP
			$\overline{2}95$		NP
105	- 5.3	0	$\overline{2},10,5$		NP
115	+ 0.2	0			
125	- 3.9	0	305	- 1.0	0
135	-14.7	13.2	315	+ 7.6	7.5
145	- 3.1	4.6	325	+ 4.8	7.5
155	- 0.6	0	335	+ 4.4	0
165	- 5.1	5.2	345	- 1.0	0
175		NP	355	+ 6.4	5.7
185		NP	365	+ 6.1	7.7
195		NP	375		NP
1,10,5		NP	385		NP
$\overline{1}05$	+ 8.9	9.7	$\overline{3}05$	-12.3	18.1
$\overline{1}15$	+ 6.4	6.0	$\overline{3}15$	- 8.6	6.0
$\overline{1}25$	+ 5.1	0	$\overline{3}25$	- 4.8	0
$\overline{1}35$	+10.3	8.3	$\overline{3}35$	- 7.4	6.0
$\overline{1}45$	+ 4.8	3.7	$\overline{3}45$	- 6.5	6.0
$\overline{1}55$	+ 6.1	4.6	$\overline{3}55$	- 7.6	0
$\overline{1}65$	+ 5.3	6.0	$\overline{3}65$	- 3.9	0
$\overline{1}75$		NP	$\overline{3}75$		NP
$\overline{1}85$		NP	$\overline{3}85$		NP
$\overline{1}95$		NP	$\overline{3}95$		NP
$\overline{1},10,5$		NP	$\overline{3},10,5$		NP



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[illegible]



TABLE I (Continued)

hk $\ell$	Calc.	Obs.	hk $\ell$	Calc.	Obs.
405	+ 5.8	0	705	- 8.0	0
415	- 1.0	0	715	- 7.1	0
425	+10.8	8.3	725	- 7.1	0
435	+ 1.2	0	735	- 2.3	0
445	- 2.8	0	745	- 1.8	0
455	- 0.1	0	755	- 5.6	0
465	+ 2.4	0	765	- 4.3	0
475		NP			
$\bar{4}05$	+15.3	20.6	$\bar{8}05$	+ 1.5	0
$\bar{4}15$	-14.1	11.5	$\bar{8}15$	+ 0.7	0
$\bar{4}25$	+ 6.4	6.0	$\bar{8}25$	+ 6.8	0
$\bar{4}35$	+ 7.3	5.5	$\bar{8}35$	- 5.5	0
$\bar{4}45$	+ 0.4	0	$\bar{8}45$	- 5.2	0
$\bar{4}55$	-11.0	0	$\bar{8}55$	+ 0.6	0
$\bar{4}65$	- 5.6	0	$\bar{8}65$	- 0.3	0
$\bar{4}75$		NP			
$\bar{4}85$		NP	$\bar{9}05$	+10.5	13.5
$\bar{4}95$		NP	$\bar{9}15$	- 0.6	0
$\bar{4}, 10, 5$		NP	$\bar{9}25$	- 0.1	0
			$\bar{9}35$	+ 6.3	0
505	- 5.6	9.5	$\bar{9}45$	+ 5.8	0
515	- 3.1	0			
525	+ 4.1	8.6	006	+ 0.7	0
535	- 4.9	3.7	016	+ 7.1	6.9
545	- 5.0	8.0	026	- 5.3	0
555	- 2.9	0	036	- 4.1	0
			046	- 5.5	0
$\bar{5}05$	+ 7.7	0	056	+ 5.6	0
$\bar{5}15$	0	0	066	-10.3	8.0
$\bar{5}25$	+ 9.1	6.0	076		NP
$\bar{5}35$	+12.5	8.6	086		NP
$\bar{5}45$	+ 2.2	0			
$\bar{5}55$	+ 0.5	0	106	+ 5.9	0
$\bar{5}65$	+ 6.2	0	116	+ 0.1	0
$\bar{5}75$		NP	126	- 2.2	0
$\bar{5}85$		NP	136	- 4.2	0
$\bar{5}95$		NP	146	+ 3.3	0
			156	+ 1.3	0
605	-14.3	12.9	166	- 3.3	0
615	+ 0.3	0	176		NP
625	- 0.7	0			
635	+ 5.7	4.3	I06	- 0.7	0
645	- 2.6	0	I16	+ 3.7	0
655	+ 0.2	0	I26	- 5.3	0
665	+ 7.5	0	I36	+ 1.8	0
			I46	+ 1.5	0

TABLE 1 (Continued)

Row	Col 1	Col 2	Col 3	Col 4	Col 5
0	0.0	0.0	0.0	0.0	0.0
0	0.1	0.1	0.1	0.1	0.1
0	0.1	0.1	0.1	0.1	0.1
0	0.2	0.2	0.2	0.2	0.2
0	0.1	0.1	0.1	0.1	0.1
0	0.2	0.2	0.2	0.2	0.2
0	0.3	0.3	0.3	0.3	0.3
0	0.4	0.4	0.4	0.4	0.4
0	0.5	0.5	0.5	0.5	0.5
0	0.6	0.6	0.6	0.6	0.6
0	0.7	0.7	0.7	0.7	0.7
0	0.8	0.8	0.8	0.8	0.8
0	0.9	0.9	0.9	0.9	0.9
0	1.0	1.0	1.0	1.0	1.0
0	1.1	1.1	1.1	1.1	1.1
0	1.2	1.2	1.2	1.2	1.2
0	1.3	1.3	1.3	1.3	1.3
0	1.4	1.4	1.4	1.4	1.4
0	1.5	1.5	1.5	1.5	1.5
0	1.6	1.6	1.6	1.6	1.6
0	1.7	1.7	1.7	1.7	1.7
0	1.8	1.8	1.8	1.8	1.8
0	1.9	1.9	1.9	1.9	1.9
0	2.0	2.0	2.0	2.0	2.0
0	2.1	2.1	2.1	2.1	2.1
0	2.2	2.2	2.2	2.2	2.2
0	2.3	2.3	2.3	2.3	2.3
0	2.4	2.4	2.4	2.4	2.4
0	2.5	2.5	2.5	2.5	2.5
0	2.6	2.6	2.6	2.6	2.6
0	2.7	2.7	2.7	2.7	2.7
0	2.8	2.8	2.8	2.8	2.8
0	2.9	2.9	2.9	2.9	2.9
0	3.0	3.0	3.0	3.0	3.0
0	3.1	3.1	3.1	3.1	3.1
0	3.2	3.2	3.2	3.2	3.2
0	3.3	3.3	3.3	3.3	3.3
0	3.4	3.4	3.4	3.4	3.4
0	3.5	3.5	3.5	3.5	3.5
0	3.6	3.6	3.6	3.6	3.6
0	3.7	3.7	3.7	3.7	3.7
0	3.8	3.8	3.8	3.8	3.8
0	3.9	3.9	3.9	3.9	3.9
0	4.0	4.0	4.0	4.0	4.0
0	4.1	4.1	4.1	4.1	4.1
0	4.2	4.2	4.2	4.2	4.2
0	4.3	4.3	4.3	4.3	4.3
0	4.4	4.4	4.4	4.4	4.4
0	4.5	4.5	4.5	4.5	4.5
0	4.6	4.6	4.6	4.6	4.6
0	4.7	4.7	4.7	4.7	4.7
0	4.8	4.8	4.8	4.8	4.8
0	4.9	4.9	4.9	4.9	4.9
0	5.0	5.0	5.0	5.0	5.0
0	5.1	5.1	5.1	5.1	5.1
0	5.2	5.2	5.2	5.2	5.2
0	5.3	5.3	5.3	5.3	5.3
0	5.4	5.4	5.4	5.4	5.4
0	5.5	5.5	5.5	5.5	5.5
0	5.6	5.6	5.6	5.6	5.6
0	5.7	5.7	5.7	5.7	5.7
0	5.8	5.8	5.8	5.8	5.8
0	5.9	5.9	5.9	5.9	5.9
0	6.0	6.0	6.0	6.0	6.0
0	6.1	6.1	6.1	6.1	6.1
0	6.2	6.2	6.2	6.2	6.2
0	6.3	6.3	6.3	6.3	6.3
0	6.4	6.4	6.4	6.4	6.4
0	6.5	6.5	6.5	6.5	6.5
0	6.6	6.6	6.6	6.6	6.6
0	6.7	6.7	6.7	6.7	6.7
0	6.8	6.8	6.8	6.8	6.8
0	6.9	6.9	6.9	6.9	6.9
0	7.0	7.0	7.0	7.0	7.0
0	7.1	7.1	7.1	7.1	7.1
0	7.2	7.2	7.2	7.2	7.2
0	7.3	7.3	7.3	7.3	7.3
0	7.4	7.4	7.4	7.4	7.4
0	7.5	7.5	7.5	7.5	7.5
0	7.6	7.6	7.6	7.6	7.6
0	7.7	7.7	7.7	7.7	7.7
0	7.8	7.8	7.8	7.8	7.8
0	7.9	7.9	7.9	7.9	7.9
0	8.0	8.0	8.0	8.0	8.0
0	8.1	8.1	8.1	8.1	8.1
0	8.2	8.2	8.2	8.2	8.2
0	8.3	8.3	8.3	8.3	8.3
0	8.4	8.4	8.4	8.4	8.4
0	8.5	8.5	8.5	8.5	8.5
0	8.6	8.6	8.6	8.6	8.6
0	8.7	8.7	8.7	8.7	8.7
0	8.8	8.8	8.8	8.8	8.8
0	8.9	8.9	8.9	8.9	8.9
0	9.0	9.0	9.0	9.0	9.0
0	9.1	9.1	9.1	9.1	9.1
0	9.2	9.2	9.2	9.2	9.2
0	9.3	9.3	9.3	9.3	9.3
0	9.4	9.4	9.4	9.4	9.4
0	9.5	9.5	9.5	9.5	9.5
0	9.6	9.6	9.6	9.6	9.6
0	9.7	9.7	9.7	9.7	9.7
0	9.8	9.8	9.8	9.8	9.8
0	9.9	9.9	9.9	9.9	9.9
0	10.0	10.0	10.0	10.0	10.0

TABLE I (Continued)

hkl	Calc.	Obs.	hkl	Calc.	Obs.
$\bar{1}56$	+ 1.5	0	$\bar{4}06$	+ 1.3	0
$\bar{1}66$	- 2.7	0	$\bar{4}16$	- 2.5	4.9
$\bar{1}76$		NP	$\bar{4}26$	- 4.2	0
$\bar{1}86$		NP	$\bar{4}36$	+ 4.6	2.9
			$\bar{4}46$	- 5.7	0
206	+ 0.8	0	$\bar{4}56$	- 1.4	0
216	+ 9.5	6.6	$\bar{4}66$	-10.0	5.2
226	+ 3.1	0	$\bar{4}76$		NP
236	-10.9	8.9	$\bar{4}86$		NP
246	+ 5.7	4.6	$\bar{4}96$		NP
256	+ 8.0	5.5			
266	+ 7.7	5.2	$\bar{5}06$	-12.2	16.6
$\bar{2}06$	+ 4.0	0	$\bar{5}16$	+ 5.1	5.5
$\bar{2}16$	- 2.9	0	$\bar{5}26$	+ 4.3	0
$\bar{2}26$	+ 0.3	0	$\bar{5}36$	+ 0.6	0
$\bar{2}36$	+ 0.1	0	$\bar{5}46$	- 9.3	0
$\bar{2}46$	+10.5	8.6	$\bar{5}56$	+ 2.8	0
$\bar{2}56$	- 2.7	0	$\bar{5}66$	+ 3.3	0
$\bar{2}66$	+ 7.4	0	$\bar{5}76$		NP
$\bar{2}76$		NP	$\bar{5}86$		NP
$\bar{2}86$		NP			
$\bar{2}96$		NP	$\bar{6}06$	+ 0.9	0
			$\bar{6}16$	- 0.1	0
306	+ 1.2	0	$\bar{6}26$	+ 0.4	0
316	+ 0.9	0	$\bar{6}36$	- 1.5	0
326	- 3.1	0	$\bar{6}46$	+ 6.4	3.7
336	+ 2.1	0	$\bar{6}56$	- 0.7	0
346	+ 3.2	3.2	$\bar{6}66$	+ 6.3	0
356	+ 0.1	0			
$\bar{3}06$	+ 2.1	0	$\bar{7}06$	- 5.5	0
$\bar{3}16$	+ 0.3	0	$\bar{7}16$	+ 3.0	0
$\bar{3}26$	+ 5.2	0	$\bar{7}26$	+10.9	4.6
$\bar{3}46$	+ 6.0	5.2	$\bar{7}36$	- 7.3	0
$\bar{3}56$	- 0.2	0	$\bar{7}46$	- 4.5	0
$\bar{3}66$	+ 1.6	0	$\bar{7}56$	+ 3.5	0
$\bar{3}76$	+ 3.2	0	$\bar{7}66$	+ 8.8	0
$\bar{3}86$		NP			
$\bar{3}96$		NP	$\bar{8}06$	+ 1.2	0
			$\bar{8}16$	+ 1.7	0
406	- 2.6	0	$\bar{8}26$	- 1.2	0
416	+ 7.4	8.9	$\bar{8}36$	- 0.5	0
426	- 0.7	0	$\bar{8}46$	- 3.2	0
436		NP			
			007	+ 8.6	7.5
			017	+ 1.9	0



TABLE 2 (Continued)

Age	Calc.	Obs.	Age	Calc.	Obs.
100	+ 1.2	0	100	+ 1.2	0
101	- 2.7	0	101	- 2.7	0
102		0	102		0
103		0	103		0
104		0	104		0
105		0	105		0
106	+ 0.0	0	106	+ 0.0	0
107	+ 0.0	0	107	+ 0.0	0
108	+ 0.0	0	108	+ 0.0	0
109	+ 0.0	0	109	+ 0.0	0
110	+ 0.0	0	110	+ 0.0	0
111	+ 0.0	0	111	+ 0.0	0
112	+ 0.0	0	112	+ 0.0	0
113	+ 0.0	0	113	+ 0.0	0
114	+ 0.0	0	114	+ 0.0	0
115	+ 0.0	0	115	+ 0.0	0
116	+ 0.0	0	116	+ 0.0	0
117	+ 0.0	0	117	+ 0.0	0
118	+ 0.0	0	118	+ 0.0	0
119	+ 0.0	0	119	+ 0.0	0
120	+ 0.0	0	120	+ 0.0	0
121	+ 0.0	0	121	+ 0.0	0
122	+ 0.0	0	122	+ 0.0	0
123	+ 0.0	0	123	+ 0.0	0
124	+ 0.0	0	124	+ 0.0	0
125	+ 0.0	0	125	+ 0.0	0
126	+ 0.0	0	126	+ 0.0	0
127	+ 0.0	0	127	+ 0.0	0
128	+ 0.0	0	128	+ 0.0	0
129	+ 0.0	0	129	+ 0.0	0
130	+ 0.0	0	130	+ 0.0	0
131	+ 0.0	0	131	+ 0.0	0
132	+ 0.0	0	132	+ 0.0	0
133	+ 0.0	0	133	+ 0.0	0
134	+ 0.0	0	134	+ 0.0	0
135	+ 0.0	0	135	+ 0.0	0
136	+ 0.0	0	136	+ 0.0	0
137	+ 0.0	0	137	+ 0.0	0
138	+ 0.0	0	138	+ 0.0	0
139	+ 0.0	0	139	+ 0.0	0
140	+ 0.0	0	140	+ 0.0	0
141	+ 0.0	0	141	+ 0.0	0
142	+ 0.0	0	142	+ 0.0	0
143	+ 0.0	0	143	+ 0.0	0
144	+ 0.0	0	144	+ 0.0	0
145	+ 0.0	0	145	+ 0.0	0
146	+ 0.0	0	146	+ 0.0	0
147	+ 0.0	0	147	+ 0.0	0
148	+ 0.0	0	148	+ 0.0	0
149	+ 0.0	0	149	+ 0.0	0
150	+ 0.0	0	150	+ 0.0	0
151	+ 0.0	0	151	+ 0.0	0
152	+ 0.0	0	152	+ 0.0	0
153	+ 0.0	0	153	+ 0.0	0
154	+ 0.0	0	154	+ 0.0	0
155	+ 0.0	0	155	+ 0.0	0
156	+ 0.0	0	156	+ 0.0	0
157	+ 0.0	0	157	+ 0.0	0
158	+ 0.0	0	158	+ 0.0	0
159	+ 0.0	0	159	+ 0.0	0
160	+ 0.0	0	160	+ 0.0	0
161	+ 0.0	0	161	+ 0.0	0
162	+ 0.0	0	162	+ 0.0	0
163	+ 0.0	0	163	+ 0.0	0
164	+ 0.0	0	164	+ 0.0	0
165	+ 0.0	0	165	+ 0.0	0
166	+ 0.0	0	166	+ 0.0	0
167	+ 0.0	0	167	+ 0.0	0
168	+ 0.0	0	168	+ 0.0	0
169	+ 0.0	0	169	+ 0.0	0
170	+ 0.0	0	170	+ 0.0	0
171	+ 0.0	0	171	+ 0.0	0
172	+ 0.0	0	172	+ 0.0	0
173	+ 0.0	0	173	+ 0.0	0
174	+ 0.0	0	174	+ 0.0	0
175	+ 0.0	0	175	+ 0.0	0
176	+ 0.0	0	176	+ 0.0	0
177	+ 0.0	0	177	+ 0.0	0
178	+ 0.0	0	178	+ 0.0	0
179	+ 0.0	0	179	+ 0.0	0
180	+ 0.0	0	180	+ 0.0	0
181	+ 0.0	0	181	+ 0.0	0
182	+ 0.0	0	182	+ 0.0	0
183	+ 0.0	0	183	+ 0.0	0
184	+ 0.0	0	184	+ 0.0	0
185	+ 0.0	0	185	+ 0.0	0
186	+ 0.0	0	186	+ 0.0	0
187	+ 0.0	0	187	+ 0.0	0
188	+ 0.0	0	188	+ 0.0	0
189	+ 0.0	0	189	+ 0.0	0
190	+ 0.0	0	190	+ 0.0	0
191	+ 0.0	0	191	+ 0.0	0
192	+ 0.0	0	192	+ 0.0	0
193	+ 0.0	0	193	+ 0.0	0
194	+ 0.0	0	194	+ 0.0	0
195	+ 0.0	0	195	+ 0.0	0
196	+ 0.0	0	196	+ 0.0	0
197	+ 0.0	0	197	+ 0.0	0
198	+ 0.0	0	198	+ 0.0	0
199	+ 0.0	0	199	+ 0.0	0
200	+ 0.0	0	200	+ 0.0	0



TABLE I (Continued)

hkl	Calc.	Obs.	hkl	Calc.	Obs.
027	+ 6.8	6.0	$\bar{4}07$	+ 7.1	9.5
037	+ 1.5	0	$\bar{4}17$	- 2.5	0
047	- 0.6	0	$\bar{4}27$	+ 9.8	5.4
057	+ 0.1	0	$\bar{4}37$	+ 3.8	0
			$\bar{4}47$	- 2.2	0
107	+ 6.4	9.8	$\bar{4}57$	- 4.0	0
117	- 4.1	0	$\bar{4}67$	+ 1.9	0
127	- 1.5	0			
137	- 4.7	3.7	507	+ 2.3	0
147	+ 2.6	0	517	- 1.0	0
			527	- 1.5	0
$\bar{1}07$	+ 4.3	0	537	+10.5	5.7
$\bar{1}17$	- 1.3	0	547	+ 2.9	0
$\bar{1}27$	- 7.7	4.3	557	- 0.8	0
$\bar{1}37$	+11.0	6.6	567	- 2.0	0
$\bar{1}47$	+ 7.3	4.9			
$\bar{1}57$	- 0.7	0	$\bar{6}07$	-10.6	11.2
$\bar{1}67$	- 5.8	0	$\bar{6}17$	- 2.8	0
			$\bar{6}27$	- 4.5	0
207	- 6.5	6.0	$\bar{6}37$	+ 2.3	0
217	- 1.3	0	$\bar{6}47$	- 1.7	0
227	- 5.3	0	$\bar{6}57$	- 0.6	0
$\bar{2}07$	-13.9	19.2	707	- 0.3	0
$\bar{2}17$	- 2.4	0	717	- 2.7	0
$\bar{2}27$	- 3.9	0	727	- 2.0	0
$\bar{2}37$	- 0.2	0	737	- 5.2	0
$\bar{2}47$	- 3.5	0			
$\bar{2}57$	- 0.2	0	$\bar{2}08$	+ 0.9	0
$\bar{2}67$	+ 3.1	0			
			$\bar{3}08$	- 3.5	0
307	+ 7.9	9.8	$\bar{3}18$	- 2.0	0
317	-10.8	9.2			
327	- 6.0	0	$\bar{4}08$	- 1.7	0
337	+ 0.3	0	$\bar{4}18$		NP
347	+ 4.2	0			
357	- 9.3	0			
367	- 4.4	0			

It is seen that general agreement is very good. Any large discrepancies may arise from poor intensity estimation

TABLE 2 (Continued)

Year	Value	Year	Value	Year	Value
1957	1.7 +	1967	1.7 +	1977	1.7 +
1958	2.2 +	1968	2.2 +	1978	2.2 +
1959	2.8 +	1969	2.8 +	1979	2.8 +
1960	3.4 +	1970	3.4 +	1980	3.4 +
1961	4.0 +	1971	4.0 +	1981	4.0 +
1962	4.6 +	1972	4.6 +	1982	4.6 +
1963	5.2 +	1973	5.2 +	1983	5.2 +
1964	5.8 +	1974	5.8 +	1984	5.8 +
1965	6.4 +	1975	6.4 +	1985	6.4 +
1966	7.0 +	1976	7.0 +	1986	7.0 +
1967	7.6 +	1977	7.6 +	1987	7.6 +
1968	8.2 +	1978	8.2 +	1988	8.2 +
1969	8.8 +	1979	8.8 +	1989	8.8 +
1970	9.4 +	1980	9.4 +	1990	9.4 +
1971	10.0 +	1981	10.0 +	1991	10.0 +
1972	10.6 +	1982	10.6 +	1992	10.6 +
1973	11.2 +	1983	11.2 +	1993	11.2 +
1974	11.8 +	1984	11.8 +	1994	11.8 +
1975	12.4 +	1985	12.4 +	1995	12.4 +
1976	13.0 +	1986	13.0 +	1996	13.0 +
1977	13.6 +	1987	13.6 +	1997	13.6 +
1978	14.2 +	1988	14.2 +	1998	14.2 +
1979	14.8 +	1989	14.8 +	1999	14.8 +
1980	15.4 +	1990	15.4 +	2000	15.4 +
1981	16.0 +	1991	16.0 +	2001	16.0 +
1982	16.6 +	1992	16.6 +	2002	16.6 +
1983	17.2 +	1993	17.2 +	2003	17.2 +
1984	17.8 +	1994	17.8 +	2004	17.8 +
1985	18.4 +	1995	18.4 +	2005	18.4 +
1986	19.0 +	1996	19.0 +	2006	19.0 +
1987	19.6 +	1997	19.6 +	2007	19.6 +
1988	20.2 +	1998	20.2 +	2008	20.2 +
1989	20.8 +	1999	20.8 +	2009	20.8 +
1990	21.4 +	2000	21.4 +	2010	21.4 +
1991	22.0 +	2001	22.0 +	2011	22.0 +
1992	22.6 +	2002	22.6 +	2012	22.6 +
1993	23.2 +	2003	23.2 +	2013	23.2 +
1994	23.8 +	2004	23.8 +	2014	23.8 +
1995	24.4 +	2005	24.4 +	2015	24.4 +
1996	25.0 +	2006	25.0 +	2016	25.0 +
1997	25.6 +	2007	25.6 +	2017	25.6 +
1998	26.2 +	2008	26.2 +	2018	26.2 +
1999	26.8 +	2009	26.8 +	2019	26.8 +
2000	27.4 +	2010	27.4 +	2020	27.4 +

It is not clear that the general agreement is very good. Any large discrepancies may arise from insufficient

and to the use of the isotropic temperature factor. The non-cylindrical cross section of the crystals used for photography increased the difficulty of good cross-calibration of intensities and may account for some errors. For the cut crystals the character of the spots left much to be desired so some deviations are to be expected for this reason. The parameters used for calculation of amplitudes were determined from observed data alone and thus final positions include no corrections for incomplete series. At high values of  $(\sin \theta)/\lambda$  very small changes in coordinates have a relatively large effect on  $F_{\text{calc}}$ . and some of the differences may be resolved by better parameter values. The determination of an  $F$  calc. synthesis is now in progress but will not be reported here.

A criterion of the quality of a structure determination has been suggested by Smare<sup>(18)</sup> and Broomhead and Nichol<sup>(4)</sup> wherein an "index of reliability" is determined as

$$R = \frac{\sum |F_{\text{obs.}}| - |F_{\text{calc.}}|}{\sum |F_{\text{obs.}}|}.$$

Applied to the data herein two values of the index were evaluated. When only reflections for which observed data are available are considered,  $R = 0.17$ . If amplitudes, the calculation of which indicates they are too weak to observe (i.e.  $< 3$ ), are omitted but all others are



and to the use of the logarithmic temperature factor.  
The non-cylindrical cross section of the crystals used  
for photography increased the difficulty of good cross-  
calibration of facettations and may account for some errors.  
For the cut crystals the character of the spots left

much to be desired as some deviations are to be expected  
for this reason. The parameters used for calculation of  
amplitudes were determined from observed data alone and  
that final position includes no corrections for inhomogene-  
ities. At high values of  $\sin \theta/\lambda$  very small changes in  
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now in progress but will not be reported here.

A criterion of the quality of a structure determina-  
tion has been suggested by <sup>(12)</sup> and <sup>(13)</sup> and <sup>(14)</sup> and <sup>(15)</sup>  
Michael<sup>(14)</sup> wherein an "index of reliability" is determined

as

$$R = \frac{\sum |F_{obs}| - \sum |F_{calc}|}{\sum |F_{obs}|}$$

Applied to the data herein two values of the index were  
evaluated. When only reflections for which observed data  
are available are considered,  $R = 0.17$ . It includes  
the calculation of which indicates they are too weak to  
observe ( $I < 3$ ), are omitted but all others are



considered, then  $R = 0.21$ . These values compare favorably with those obtained in other structure determinations by other investigators. More recently it has been suggested that the index is not as good an indication of the quality of a structure as originally supposed.

The quality of a structure is originally supposed to be good. The index is not a good indication of the quality of a structure. It has been suggested that the index be used as a guide to the quality of a structure. The index is not a good indication of the quality of a structure. It has been suggested that the index be used as a guide to the quality of a structure.

## DISCUSSION OF THE STRUCTURE

As has been stated, there are two strings of molecules, one in the plane  $y = 1/4$  and the other in  $y = 3/4$  so oriented with respect to each other as to satisfy best packing relations. Distances of close approach in the cell have been calculated and are shown in Figure 9.

To simplify the discussion of the molecular structure the atoms of the molecule have been labelled in Figure 2. Figure 10 is a sketch showing distances and angles between the atoms lying in the symmetry plane. A similar sketch for the atoms occupying fourfold positions is shown in Figure 11. Table II below lists pertinent intramolecular distances and angles together with average values of interest.

TABLE II  
(All Distances in Angstroms)

C <sub>1</sub> - Cl <sub>7</sub>	1.80	Cl <sub>1</sub> - Cl <sub>4</sub>	2.81
C <sub>1</sub> - Cl <sub>6</sub>	1.67	Cl <sub>5</sub> - Cl <sub>8</sub>	2.84
C <sub>3</sub> - Cl <sub>8</sub>	1.75	Cl <sub>6</sub> - Cl <sub>7</sub>	2.82
C <sub>3</sub> - Cl <sub>5</sub>	1.82	Average	2.82
C <sub>2</sub> - Cl <sub>1</sub>	1.75	Cl <sub>7</sub> - Cl <sub>8</sub>	4.93
C <sub>2</sub> - Cl <sub>4</sub>	1.71	Cl <sub>3</sub> - Cl <sub>4</sub>	4.92
Average	1.75	Average	4.92

As has been stated, there are two regions of order, one in the plane  $y = 1/2$  and the other in  $y = 1/4$ . In oriented with respect to each other as is easily seen from Figure 1. Distances of lines measured in the cell have been calculated and are shown in Figure 2. To simplify the discussion of the molecular structure the atoms of the molecule have been labeled in Figure 1. Figure 1 is a sketch showing distances and angles between the atoms lying in the symmetry plane, a similar sketch for the atoms occupying tetrahedral positions is shown in Figure 11. Table II below lists positions of atoms and angles together with average values of interest.

TABLE II  
(All distances in Angstroms)

$C_1 - C_2$	1.35	$C_1 - C_3$	1.35
$C_1 - C_4$	1.35	$C_1 - C_5$	1.35
$C_2 - C_3$	1.35	$C_2 - C_4$	1.35
$C_2 - C_5$	1.35	$C_3 - C_4$	1.35
$C_3 - C_5$	1.35	$C_4 - C_5$	1.35
$C_1 - C_6$	1.35	$C_1 - C_7$	1.35
$C_1 - C_8$	1.35	$C_1 - C_9$	1.35
$C_2 - C_6$	1.35	$C_2 - C_7$	1.35
$C_2 - C_8$	1.35	$C_2 - C_9$	1.35
$C_3 - C_6$	1.35	$C_3 - C_7$	1.35
$C_3 - C_8$	1.35	$C_3 - C_9$	1.35
$C_4 - C_6$	1.35	$C_4 - C_7$	1.35
$C_4 - C_8$	1.35	$C_4 - C_9$	1.35
$C_5 - C_6$	1.35	$C_5 - C_7$	1.35
$C_5 - C_8$	1.35	$C_5 - C_9$	1.35
$C_6 - C_7$	1.35	$C_6 - C_8$	1.35
$C_6 - C_9$	1.35	$C_7 - C_8$	1.35
$C_7 - C_9$	1.35	$C_8 - C_9$	1.35
$C_1 - C_{10}$	1.35	$C_1 - C_{11}$	1.35
$C_1 - C_{12}$	1.35	$C_1 - C_{13}$	1.35
$C_2 - C_{10}$	1.35	$C_2 - C_{11}$	1.35
$C_2 - C_{12}$	1.35	$C_2 - C_{13}$	1.35
$C_3 - C_{10}$	1.35	$C_3 - C_{11}$	1.35
$C_3 - C_{12}$	1.35	$C_3 - C_{13}$	1.35
$C_4 - C_{10}$	1.35	$C_4 - C_{11}$	1.35
$C_4 - C_{12}$	1.35	$C_4 - C_{13}$	1.35
$C_5 - C_{10}$	1.35	$C_5 - C_{11}$	1.35
$C_5 - C_{12}$	1.35	$C_5 - C_{13}$	1.35
$C_6 - C_{10}$	1.35	$C_6 - C_{11}$	1.35
$C_6 - C_{12}$	1.35	$C_6 - C_{13}$	1.35
$C_7 - C_{10}$	1.35	$C_7 - C_{11}$	1.35
$C_7 - C_{12}$	1.35	$C_7 - C_{13}$	1.35
$C_8 - C_{10}$	1.35	$C_8 - C_{11}$	1.35
$C_8 - C_{12}$	1.35	$C_8 - C_{13}$	1.35
$C_9 - C_{10}$	1.35	$C_9 - C_{11}$	1.35
$C_9 - C_{12}$	1.35	$C_9 - C_{13}$	1.35
$C_{10} - C_{11}$	1.35	$C_{10} - C_{12}$	1.35
$C_{10} - C_{13}$	1.35	$C_{11} - C_{12}$	1.35
$C_{11} - C_{13}$	1.35	$C_{12} - C_{13}$	1.35
$C_{10} - C_{14}$	1.35	$C_{10} - C_{15}$	1.35
$C_{10} - C_{16}$	1.35	$C_{10} - C_{17}$	1.35
$C_{11} - C_{14}$	1.35	$C_{11} - C_{15}$	1.35
$C_{11} - C_{16}$	1.35	$C_{11} - C_{17}$	1.35
$C_{12} - C_{14}$	1.35	$C_{12} - C_{15}$	1.35
$C_{12} - C_{16}$	1.35	$C_{12} - C_{17}$	1.35
$C_{13} - C_{14}$	1.35	$C_{13} - C_{15}$	1.35
$C_{13} - C_{16}$	1.35	$C_{13} - C_{17}$	1.35
$C_{14} - C_{15}$	1.35	$C_{14} - C_{16}$	1.35
$C_{14} - C_{17}$	1.35	$C_{15} - C_{16}$	1.35
$C_{15} - C_{17}$	1.35	$C_{16} - C_{17}$	1.35



TABLE II (Continued)

Cl <sub>5</sub> - Cl <sub>6</sub>	3.36	Cl <sub>4</sub> - Cl <sub>7</sub>	3.96
Cl <sub>1</sub> - Cl <sub>2</sub>	3.38	Cl <sub>4</sub> - Cl <sub>8</sub>	4.00
Average	3.37	Average	3.98
C <sub>1</sub> - C <sub>3</sub>	2.12	∠ Cl <sub>1</sub> -C <sub>2</sub> -C <sub>4</sub>	108° - 20'
C <sub>2</sub> - C <sub>4</sub>	2.21	∠ Cl <sub>6</sub> -C <sub>1</sub> -Cl <sub>7</sub>	108° - 20'
Average	2.16	∠ Cl <sub>5</sub> -C <sub>3</sub> -Cl <sub>8</sub>	106°
		Average	107° - 33'
C <sub>1</sub> - C <sub>2</sub>	1.59		
C <sub>3</sub> - C <sub>2</sub>	1.55	∠ C <sub>1</sub> -C <sub>2</sub> -C <sub>3</sub>	85°
Average	1.57	∠ C <sub>2</sub> -C <sub>1</sub> -C <sub>4</sub>	88°
		Average	86°.5
Cl <sub>4</sub> - Cl <sub>6</sub>	3.06		
Cl <sub>4</sub> - Cl <sub>5</sub>	3.10		
Cl <sub>1</sub> - Cl <sub>7</sub>	3.11		
Cl <sub>1</sub> - Cl <sub>8</sub>	3.07		
Average	3.08		

The twofold chlorine atoms form an isosceles trapezoid lying in the symmetry plane and the chlorines occupying fourfold positions form a similar trapezoid essentially of the same dimensions which lies almost in the plane  $x = 1/4$ . The two trapezoids are so oriented as to give the idealized molecule the symmetry  $\bar{4}n$ . From an inspection of the Cl - Cl separations on each side of



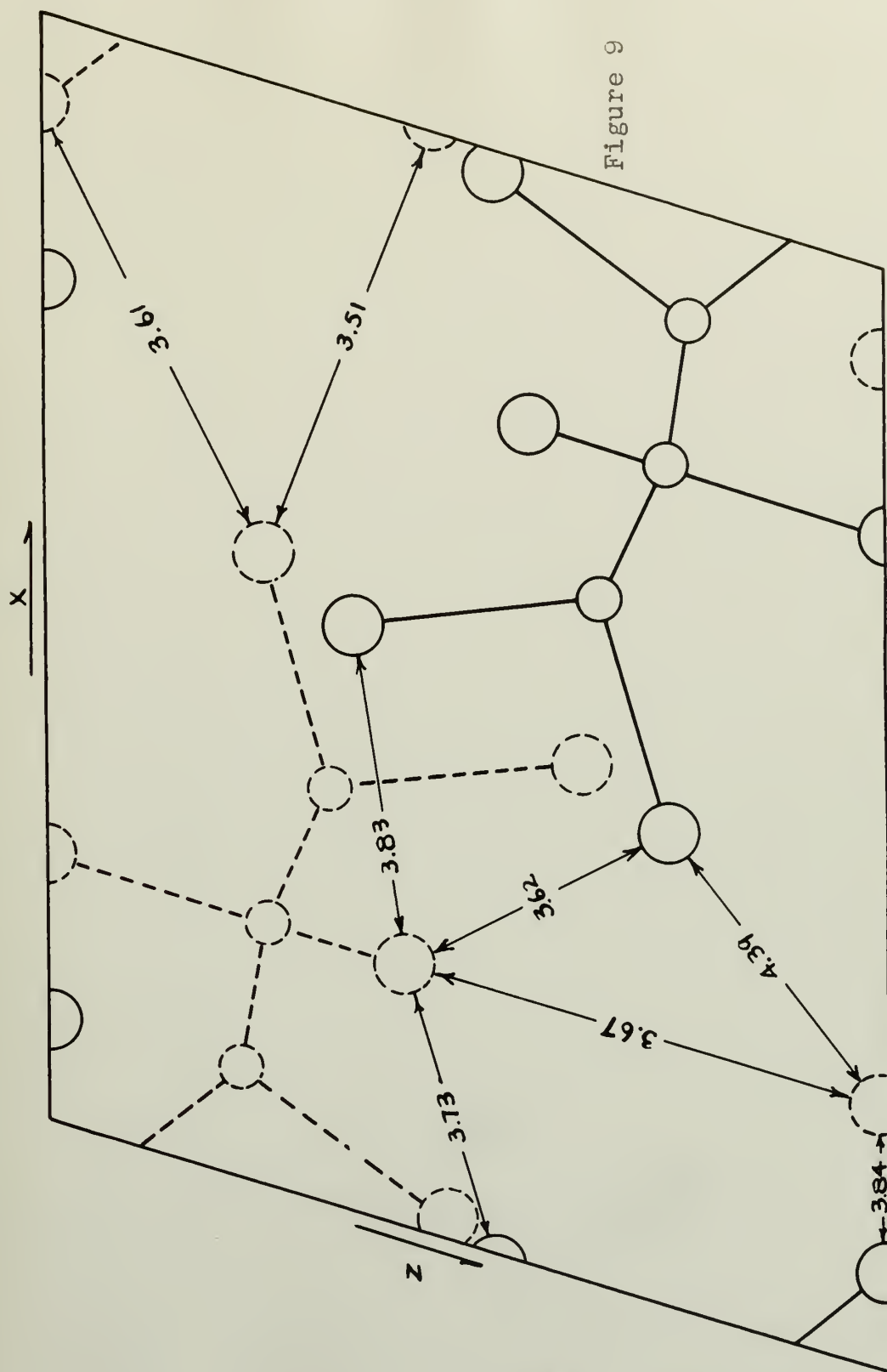
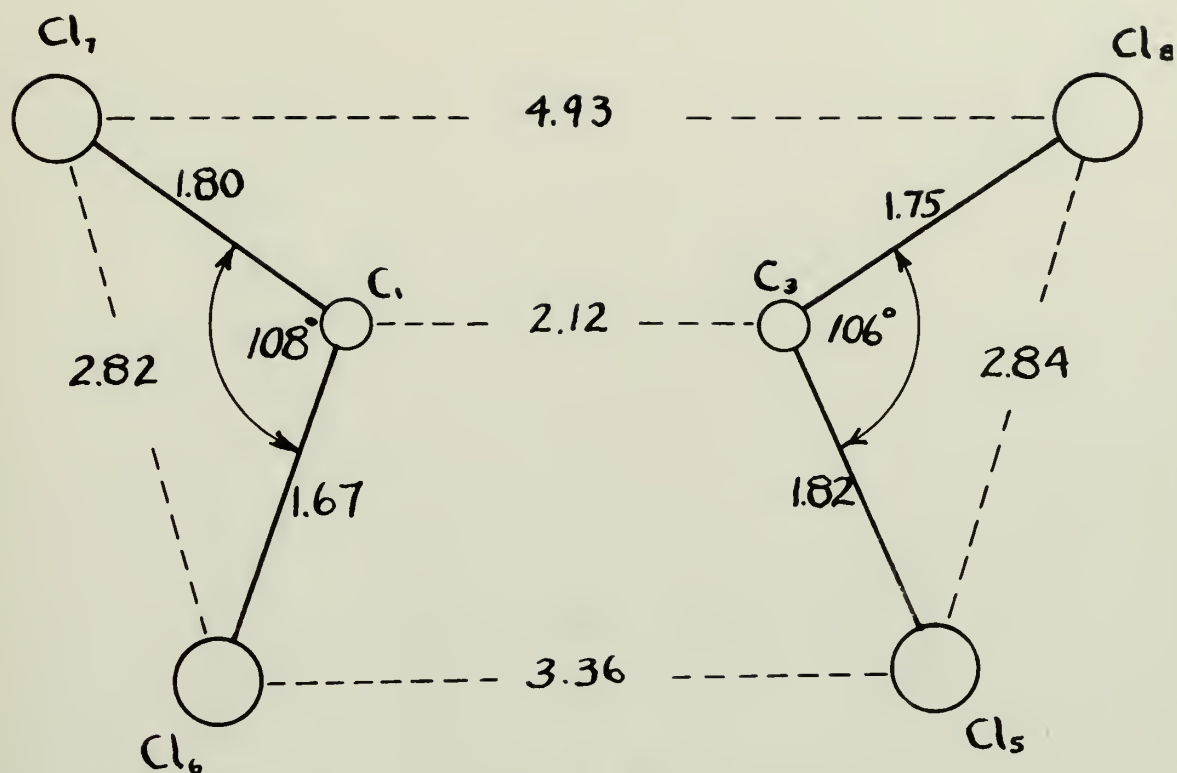


Figure 9

SKETCH SHOWING SOME VAN DER WAAL SEPARATIONS







DISTANCES IN PLANE  $Y = \frac{1}{4}$

Figure 10



the carbon ring it can be seen that the chlorines have moved so as to equalize Cl - Cl interactions at the expense of deformation of the carbon ring. It is felt that the small amount of energy required for distortion of the ring is more than regained by equalization of the chlorine distances.

The optical behavior of the compound is of interest because the crystals show extinction parallel to the needle axis which the study has shown not to be the unique axis. In the monoclinic case, the orientation of the indicatrix must conform to the crystal symmetry and one axis thereof must coincide with the b axis of the unit cell. Ordinarily this is the only restriction on indicatrix orientation except that its other two axis must obviously lie in the ac plane taking any position in this plane but of course remaining at right angles to each other. For  $C_4Cl_8$  the conclusion is that one of the two indicatrix axes in the ac plane lies parallel to c. This might be expected since the strings of molecules also lie parallel to c as does the fourfold alternating axis of the idealized molecule. The remaining indicatrix axis must then lie normal to c. It is believed that this optical behavior is good evidence in support of the conclusion that the molecular symmetry approximates closely to  $\bar{4}m$ .

The common ring is can be seen that the distances have moved on as the angles  $\alpha$  -  $\beta$  increase at the expense of deformation of the carbon ring. It is said that the small amount of energy required for distortion of the ring is more than regained by equalization of the valence distances.

The optical behavior of the compound is of interest because the crystals show extinction parallel to the  $ac$  axis which the study has shown not to be the unique axis. In the monoclinic case, the extinction of the indicatrix must conform to the crystal symmetry and one axis necessarily must coincide with the  $b$  axis of the unit cell. Ordinarily this is the only restriction on indicatrix extinction except that the other two axes must necessarily lie in the  $ac$  plane taking any position in this plane but of course remaining at right angles to each other. But taking the convention is that one of the two indicatrices lies in the  $ac$  plane also parallel to  $b$ . This might be expected since the strings of molecules also lie parallel to  $b$  as does the  $b$  axis of alternating axis of the lattice molecule. The remaining indicatrix axis must then lie normal to  $b$ . It is believed that this optical behavior is good evidence in support of the conclusion that the molecular symmetry approximates closely to  $C_{2v}$ .



The puckering of the carbon ring is considerable, the dihedral angle being about  $16^\circ$ . From an examination of the literature this appears to be the first direct evidence of a non-planar cyclobutane ring. Edgell<sup>(10)</sup> from the infra-red and Raman spectra study of  $C_4F_8$  concludes that the ring is planar with a molecular symmetry  $D_{4h}$ . Preliminary results of electron diffraction studies of the same molecule by Livingston indicate that the ring is puckered. Spectroscopic studies of  $C_4H_8$  are interpreted by Wilson<sup>(20)</sup> to show the molecular symmetry is not  $C_{2v}$  but rather  $D_{4h}$ . Dunitz' X-ray study of tetraphenyl cyclobutane shows the ring carbons to lie in a single plane, a symmetry center for the molecule being required for the space group.

Individual C-Cl distances are erratic because of poor carbon peaks in the Fourier sections. However they average out to  $1.75 \text{ \AA}$ , which is very close to the distance of  $1.76 \text{ \AA}$  observed by Pauling and Brockway<sup>(15)</sup> in electron diffraction studies of  $CCl_4$ . In liquid carbon tetrachloride Bray and Gingrich<sup>(3)</sup> have observed the corresponding value as  $1.74 \text{ \AA}$ . For chlorines bonded to the same carbon atom an average separation of  $2.82 \text{ \AA}$  is found in  $C_4Cl_8$ . A value of  $2.86 \text{ \AA}$  is reported by Pauling and Brockway as well as by Cosslett and de Laszlo<sup>(7)</sup> in independent investigations of gaseous  $CCl_4$ .

The presence of the carbon ring is considerable.

The observed angle being about  $10^\circ$ , there is considerable

of the literature this appears to be the first direct

evidence of a non-planar cyclobutane ring. (10)

From the infrared and Raman spectra study of  $C_4H_8$  con-

cluded that the ring is planar with a molecular symmetry

$D_{2h}$ . (11) Recently results of electron diffraction studies

of the same molecule by LeFevre and co-workers (12) and the ring

is planar. Spectroscopic studies of  $C_4H_8$  and isomer-

ed by Wilson (13) as well as molecular symmetry is not

$C_{2v}$  but rather  $D_{2h}$ . Further X-ray study of tetramethyl-

ethylene shows the ring carbon to lie in a plane

plane, a symmetry center for the molecule being required

for the space group.

Deductive C-Cl distances are similar because of

short carbon bonds in the Fourier synthesis. However they

average out to  $1.75 \text{ \AA}$ . which is very close to the distance

of  $1.76 \text{ \AA}$ . observed by Felling and Hildebrand (14) in elec-

tron diffraction studies of  $CCl_4$ . In liquid carbon

tetrachloride gas, the distances (15) have been observed as

corresponding values as  $1.76 \text{ \AA}$ . for chlorine bonded to

the same carbon atom an average separation of  $2.67 \text{ \AA}$ .

is found in  $CCl_4$ . A value of  $2.66 \text{ \AA}$ . is reported by

Felling and Hildebrand as well as by Conner and de Waele (17)

in independent investigations of gaseous  $CCl_4$ .



Eisenstein<sup>(11)</sup> found the separation to be  $2.95 \text{ \AA}$ . at  $27^{\circ}\text{C}$  from X-ray studies for the liquid. The Cl-C-Cl angles observed in  $\text{C}_4\text{Cl}_8$  were all slightly less than tetrahedral.

Carbon-Carbon bond lengths are not too accurate but the average value is indicative of a departure from the "normal" value of  $1.54 \text{ \AA}$ . The following table summarizes C-C spacings in compounds containing cyclobutane rings. In every case the values are greater than the normal value.

<u>Investigator</u>	<u>Compound</u>	<u>C-C</u> ( $\text{\AA}$ .)
Dunitz	Tetraphenyl-cyclobutane	$1.585 \pm 0.02$ $1.555 \pm 0.02$ } Avg. 1.57
Bauer & Beach <sup>(1)</sup>	Methylene	$1.56 \pm 0.03$
	(17) cyclobutane	
Shand, Schomaker, & Fischer	Methylene	$1.55 \pm 0.02$
	cyclobutane	
Lipscomb and Schomaker <sup>(13)</sup>	Dimethyl ketene dimer	$1.56 \pm 0.05$
Livingston	Perfluoro-cyclobutane ( $\text{C}_4\text{F}_8$ )	1.60
This investigation	$\text{C}_4\text{Cl}_8$	$1.59$ $1.55$ } Avg. 1.57

Preliminary X-ray analysis of dinaphthylene cyclobutane by Dunitz and Weissman<sup>(9)</sup> indicate that C-C distances in the central ring may be longer than  $1.54 \text{ \AA}$ . also. They report that the molecule has a center of symmetry which means the ring must be planar.

The average C-C-C angle of  $86.5^{\circ}$  is of course only indicative of the puckering of the ring and cannot be taken as a final value since more accurate carbon positions are yet to be obtained.





## SUGGESTIONS FOR FURTHER STUDY

In any refinement procedure one may employ, the atomic coordinates of the chlorine atoms will change little if at all. There does remain the problem of tiling down the positions of the carbon atoms. There are methods that may be used to improve the structure determination.

It has been mentioned previously that many relatively intense reflections were observed at high values of  $(\sin \theta)/\lambda$ . This indicates that probably there are numerous amplitudes of considerable magnitude beyond the maximum angle observable with Cu  $K\alpha$  radiation but which still would contribute substantially to the Fourier summation. The incomplete series gives rise to a rippling effect which results in a displacement of maxima, particularly those of carbon atoms, from their true positions. It is desirable to obtain additional terms in the series to smooth out these ripples. Photographs taken with  $Mg$   $K\alpha$  radiation should serve to pick up reflections further out in the reciprocal lattice. Incorporation of those additional amplitudes in the Fourier series should then lead to more accurate positions for carbon atoms.

There is a less desirable procedure that may be

In any refinement procedure one may suppose the  
 would maintain the relative areas will change  
 little if at all. There are cases the position of  
 lines when the position of the main area. There  
 the results that may be used to improve the  
 resolution.

It has been mentioned previously that very relatively  
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 sibly from the center of the area, from their true posi-  
 tions. It is desirable to obtain additional data in  
 the series to correct for these effects. Photographs  
 taken with a  $\lambda$  radiation should serve to pick up the  
 positions further out in the reciprocal lattice. Inter-  
 position of these additional amplitudes in the Fourier  
 series should lead to more accurate positions for  
 lattice planes.

There is a less desirable procedure that may be



followed but which is considered adequate by many in the field. The parameter values given by the Fourier synthesis of observed amplitudes are used to calculate structure amplitudes for the observed range of  $(\sin \theta)/\lambda$ . These calculated amplitudes are put through a Fourier synthesis which, since this series also is somewhat incomplete, will not reproduce exactly the peak positions used to obtain the calculated Fourier coefficients. The resulting small shift in position of each peak is applied with reversed sign as a correction to the corresponding atomic positions as given by the experimental Fourier synthesis.

Since carbon maxima may be displaced and ill-defined in the presence of the high chlorine peaks, it would be desirable if the Fourier syntheses could be calculated in such a way that the contributions of the chlorines to the series would not be included. Such a procedure was partially successful in the structure determination of  $\text{UF}_6$  in the sense that when uranium peaks were subtracted out, rather well defined peaks for the light fluorine atoms remained. The method of attack was first to determine a scattering factor curve for uranium. Then structure factors were calculated for the U atoms alone and subtracted from the observed values. This left a residue which was taken as the contribution from fluorine atoms.

calculated and which is considered adequate by many in the field. The parameter values given by the Fourier synthesis of observed amplitudes are used to calculate observed amplitudes for the observed range of  $\lambda$ . These calculated amplitudes are put through a Fourier synthesis which, above this range, also is somewhat incomplete, will not reproduce exactly the peak positions used to obtain the calculated Fourier coefficients. The resulting small shift in position of each peak is applied with reversed sign as a correction to the corresponding atomic positions as given by the experimental Fourier synthesis.

Since known metals may be disordered and ill-defined in the presence of the high electron density, it would be desirable if the Fourier synthesis could be calculated in such a way that the contributions of the disorder to the series would not be included. Such a procedure was partially successful in the structure determination of  $U_3O_8$  in the sense that when certain peaks were subtracted out, better well defined peaks for the light fluorine atoms remained. The series of  $U_3O_8$  was first to determine a scattering factor curve for uranium. Then atomic factor were calculated for the  $U$  atoms alone and subtracted from the observed values. This left a residual which was taken as the contribution from fluorine atoms.



Fourier calculations with the residue gave light atom peaks and enabled the investigators to obtain approximate atomic coordinates. This same procedure could probably be carried out for  $C_4Cl_8$  but the determination of the experimental form factor for chlorine is at best a rather uncertain procedure.

positive relationship with the results gave little room  
 for the results to indicate the importance of certain aspects  
 of the results. This same procedure should  
 probably be carried out for the other two experiments  
 of the experimental design. The results of the first  
 a rather small number of subjects.

PART II

FURTHER STUDY OF THE STRUCTURE OF  $C_8F_{12}$





## INTRODUCTION

The first structure work on the dimer of hexafluorobutadiene,  $C_8F_{12}$ , was done by Dr. R. W. Broge<sup>(21)</sup> in this Laboratory. The trial model employed was based on a "chair" configuration of the three cyclobutane rings fused at  $120^\circ$  angles to one another. Reasons for the choice of such a model are reviewed in his work. He found the unit cell to be triclinic and to simplify the problem based his analysis on a face-centered triclinic unit of  $\underline{a_1}' = 9.95 \text{ \AA.}$   $\alpha' = 86.2^\circ$

$$\underline{a_2}' = 7.39 \text{ \AA.} \quad \beta' = 84.8^\circ$$

$$\underline{a_3}' = 13.20 \text{ \AA.} \quad \gamma' = 86.8^\circ$$

containing one molecule per lattice point. Since no positive pyroelectric effect was observed the molecule was believed to have a center of symmetry. Using zero-layer  $a'$  - axis data with the centrosymmetric model he determined a set of atomic parameters which, however, led to erratic C-C and C-F bond lengths and to poor agreement between calculated and observed amplitudes. It was clear that the structure problem required further study.

Since for triclinic lattices the choice of a

The first structure work on the alkyl of base-  
 fluorosulfonates,  $\text{C}_2\text{F}_5\text{SO}_3\text{H}$ , was done by Dr. E. J. Broyer  
 in this laboratory. The vital model synthesis was based  
 on a 'chain' configuration of the three substituents  
 rings fused at  $120^\circ$  angles to one another, persons  
 for the choice of such a model are reviewed in his work.  
 He found the model call to be realistic and so slightly  
 the problem based his analysis on a face-centered cub-

$$\begin{aligned} \frac{a_1}{a_2} &= 0.95 \text{ \AA.} & \lambda' &= 0.95^\circ \\ \frac{a_2}{a_3} &= 1.25 \text{ \AA.} & \delta' &= 94.5^\circ \\ \frac{a_3}{a_4} &= 1.35 \text{ \AA.} & \gamma' &= 98.5^\circ \end{aligned}$$

containing one molecule per lattice point. Since no  
 positive pyroelectric effect was observed the molecule  
 was believed to have a center of symmetry. Using info-  
 layer  $\alpha'$  - data only with the centrosymmetric model he  
 determined a set of atomic parameters which, however,  
 led to electric C-S and C-F bond lengths and to poor  
 agreement between calculated and observed amplitudes.  
 It was clear that the structure problem required further  
 study.

Since the previous studies the choice of a

structural unit is not indicated by symmetry, Buerger has recommended that such lattices be reported in terms of what he calls the "reduced primitive cell." This is a unit the edges of which have the three shortest translations in the lattice and the axes of which are, labelled such that  $\underline{a}_1 < \underline{a}_2 < \underline{a}_3$ . An additional requirement is that the interaxial angles  $\alpha$ ,  $\beta$ , and  $\gamma$  are all obtuse. It was thought that if all reflections that might be observed were indexed on the basis of this unit the problem might be clarified and some progress might be made toward the final solution.

Figure 12 shows the face centered unit used by Broge together with the primitive reduced cell employed in the present study. It is seen that  $\underline{a}_1 = \text{BD}$ ,  $\underline{a}_2 = \text{DE}$ , and  $\underline{a}_3 = \text{AB}$ . The transformation from the compound to the primitive unit is:

$$\underline{a}_1 = \frac{-\underline{a}_2' + \underline{a}_1'}{2}$$

$$\underline{a}_2 = \frac{-\underline{a}_1' - \underline{a}_2'}{2}$$

$$\underline{a}_3 = \frac{-\underline{a}_3' + \underline{a}_2'}{2}$$



structural unit is not indicated by symmetry. However, her recommendations that such lattices be treated in terms of what we call the "reduced primitive cell." This is a unit the edges of which have the same direction as the edges of the lattice and the area of which is equal to the area of the lattice. An additional negative result is that the internal angles  $\alpha, \beta, \gamma$  are all obtuse. It was thought that if all reflections that might be observed were indexed on the basis of this unit the problem might be simplified and some progress might be made toward the final solution.

Figure 12 shows the face centered unit used by Bragg together with the primitive reduced cell employed in the present study. It is seen that  $a_1 = 2a$ ,  $a_2 = 2a$ , and  $a_3 = 2a$ . The transformation from the compound to the primitive unit is:

$$\frac{a_1}{2} = a, \quad \frac{a_2}{2} = a, \quad \frac{a_3}{2} = a$$

$$\frac{a_1}{2} = a, \quad \frac{a_2}{2} = a, \quad \frac{a_3}{2} = a$$

$$\frac{a_1}{2} = a, \quad \frac{a_2}{2} = a, \quad \frac{a_3}{2} = a$$



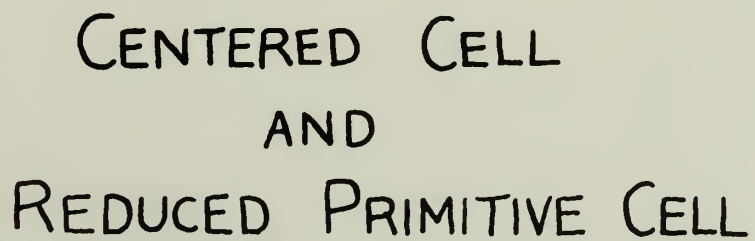


Figure 12



The present work was based on the centrosymmetric model. Although practically complete data for the primitive cell were obtained, no new atomic coordinates have been assigned and the structure still awaits solution. It is to be understood that the work reported herein was done prior to the investigation of the structure of octochlorocyclobutane. In view of the fact that the carbon ring is puckered in  $C_4Cl_8$ , it may be that the rings in  $C_8F_{12}$  are not planar. If this be true a new trial model having no center of symmetry must be considered.

The present work was based on the centrosymmetric

model. Although practically complete data for the primitive cell were obtained, no new atomic coordinates have been assigned and the structure still awaits solution. It is to be understood that the work reported

herein was done prior to the investigation of the structure of  $\text{CaSO}_4 \cdot \frac{1}{2}\text{H}_2\text{O}$ . In view of the fact that the carbon atom is positioned in  $\text{CaSO}_4$ , it may be that the atoms in  $\text{CaSO}_4$  are not planar. If this be true a new trial model having no center of symmetry must be considered.



## EXPERIMENTAL PROCEDURE

Single crystals of the compound were grown under vacuum by sublimation in thin-walled glass capillaries. The procedure is described by Broge and is a standard procedure in this Laboratory for crystals having high vapor pressures. In this study the capillaries were about 1 mm. in diameter and wall thickness averaged 0.01 mm. as measured under a microscope fitted with a micrometer eyepiece.

The crystals were mounted on the goniometer head such that the face of growth in the capillary was perpendicular to the rotation axis. Oscillation photographs were used to locate rational directions and to align the crystals for Weissenberg photography. Since the various repeat distances for rational directions could be calculated from Broge's work it was a simple matter to identify these directions on the photographs. Four crystals were photographed and the rational directions found may be described as follows:

<u>Crystal number</u>	<u>Repeat distance (<math>\text{\AA}.</math>)</u>	<u>Description</u>
1	6.3	$\underline{a_2}$ of reduced unit
2	6.0	$\underline{a_1}$ of reduced unit
3	6.3	$\underline{a_2}$ of reduced unit
4	7.4	$\underline{a_2}$ of centered cell.

The single crystals of the polymer were grown under vacuum by annealing in thin-walled glass capillaries. The procedure is described by Stoye and his students in this laboratory for systems having high vapor pressure. In this study the capillaries were about 1 mm. in diameter and well shrouded except for a 0.01 mm. diameter central microscope lens with a minimized aperture. The crystals were mounted on the condenser head such that the face of growth in the capillary was perpendicular to the rotation axis. Oscillation photographs were used to locate rotational directions and to align the crystals for x-ray diffraction photography. Since the various repeat distances for rotational directions could be calculated from Stoye's work it was a simple matter to identify these directions on the photographs. Four crystals were photographed and the rotational directions found may be described as follows:

<u>Crystal number</u>	<u>Repeat distance (Å.)</u>	<u>Orientation</u>
1	6.3	$\frac{1}{2}$ of reduced unit
2	6.0	$\frac{1}{2}$ of reduced unit
3	6.3	$\frac{1}{2}$ of reduced unit
4	7.4	$\frac{1}{2}$ of reduced unit

Crystal #1 was cracked and gave separation of spots; it was discarded after #3 was obtained.

Complete zero and higher layer equi-inclination Weissenberg photographs of all samples were taken with Cu K $\alpha$  radiation using the triple-film technique. The films were given exposure times of thirty minutes and four hours for each layer in order to be able to read spots over a wide range of intensity. Zero-layer NaCl reflections were superimposed on separate zero-layer photographs of C<sub>8</sub>F<sub>12</sub> for good lattice constant measurement.

#### Indexing the Reflections.

The problem was simplified in that the reciprocal lattice of the primitive cell was known. In that lattice it was thus possible to calculate distances and angles which served to identify unequivocally the reflections on the films. Reflections from crystals 1, 2, and 3 were easily indexed. For crystal #4 it was necessary to choose an appropriate reciprocal lattice and the corresponding direct cell for use in indexing reflections and then transform indices so that they would refer to the reduced unit. The cell chosen was:

$$\begin{array}{ll} \underline{a_1}'' = 6.03 \text{ \AA} & \alpha'' = 57^\circ \\ \underline{a_2}'' = 7.39 & \beta'' = 74^\circ \\ \underline{a_3}'' = 7.76 & \gamma'' = 55.5^\circ \end{array}$$



Circuit A1 was checked and gave separation of spots.  
It was discarded after it was obtained.

Complete sets and higher order reflections  
were photographed at all angles with  
Cu K $\alpha$  radiation using the typical technique. The  
films were given exposure times of thirty minutes and  
four hours for each layer in order to be able to scan  
spots over a wide range of intensity. Two-layer and  
reflections were superimposed on separate two-layer  
photographs of type for good lattice constant measure-  
ment.

Indexing the reflections.

The problem was simplified in that the reciprocal  
lattice of the primitive cell was known. In that lattice  
it was thus possible to calculate distances and angles  
which served to identify unambiguously the reflections  
on the films. Reflections from crystals 1, 2, and 3  
were easily indexed. For crystal 4 it was necessary  
to choose an appropriate reciprocal lattice and the cor-  
responding direct cell for use in indexing reflections and  
then translate indices so that they would refer to the  
reduced unit. The cell chosen was:

$$\begin{aligned} \underline{a}_1'' &= 6.01 \text{ \AA} \\ \underline{a}_2'' &= 7.25 \\ \underline{a}_3'' &= 7.75 \end{aligned}$$



The transformation to the primitive cell is:

$$\underline{a}_1 = \underline{a}_1''$$

$$\underline{a}_2 = \underline{a}_2'' - \underline{a}_1''$$

$$\underline{a}_3 = \underline{a}_3'' - \underline{a}_2'' .$$

Determination of the Lattice constants.

Using the cell constants determined by Broge for the centered unit the reciprocal lattice for the reduced cell has

$$\underline{a}_1^* = 0.1891 \text{ \AA}^{-1} \quad \alpha^* = 70.8^\circ$$

$$\underline{a}_2^* = 0.1742 \quad \beta^* = 66.3^\circ$$

$$\underline{a}_3^* = 0.1525 \quad \gamma^* = 68.0^\circ$$

However measurements on zero-layer Weissenberg photographs taken in the present study lead to

$$\underline{a}_1^* = 0.1890 \pm 0.0002 \text{ \AA}^{-1} \quad \alpha^* = 69.3^\circ$$

$$\underline{a}_2^* = 0.1772 \pm 0.0002 \quad \beta^* = 66.3^\circ$$

$$\underline{a}_3^* = 0.1545 \pm 0.0002$$

The spacing of  $\underline{a}_2$  was measured from an oscillation photograph upon which NaCl reflections had been superimposed and was found to be  $6.29 \text{ \AA}$ . Using this value with appropriate formulae the reciprocal lattice angle  $\gamma^*$

The transmission to the positive cell is

$$\frac{I_+}{I_0} = \frac{1}{2} \left( 1 + \frac{1}{2} \right)$$

$$\frac{I_-}{I_0} = \frac{1}{2} \left( 1 - \frac{1}{2} \right)$$

$$\frac{I_+}{I_0} = \frac{1}{2} \left( 1 + \frac{1}{2} \right)$$

Determination of the lattice constants.

Using the cell constants determined by X-ray the scattering angle the reciprocal lattice for the reduced

cell has

$$\frac{a_1^*}{a_1} = 0.1001 \pm 0.0001$$

$$\frac{a_2^*}{a_2} = 0.1001 \pm 0.0001$$

$$\frac{a_3^*}{a_3} = 0.1001 \pm 0.0001$$

However measurements on zero-layer reflexions photographs taken in the present study lead to

$$\frac{a_1^*}{a_1} = 0.1002 \pm 0.0002$$

$$\frac{a_2^*}{a_2} = 0.1002 \pm 0.0002$$

$$\frac{a_3^*}{a_3} = 0.1002 \pm 0.0002$$

The spacing of  $a_3^*$  was measured from an oscillation photo-

graph upon which well reflections had been superimposed

and was found to be  $0.1002 \pm 0.0002$ . Using this value with

appropriate formulae the reciprocal lattice angle  $\gamma^*$

was determined to be  $67.4^\circ$ . Direct cell constants are then

$$\underline{a_1} = 6.02 \text{ \AA} \quad \alpha = 103.6^\circ$$

$$\underline{a_2} = 6.29 \quad \beta = 107.9^\circ$$

$$\underline{a_3} = 7.27 \quad \gamma = 106.4^\circ$$

for the primitive reduced unit. These values lead to a unit cell volume of  $235.2 \text{ \AA}^3$  and an X-ray density of  $2.29 \text{ gm./cc.}$  Broge lists  $240.7 \text{ \AA}^3$  and  $2.23 \text{ gm./cc.}$  as a result of his study. It is to be noted that he had to employ the method of angular lag to determine angles in reciprocal space.

#### Intensity Measurement.

The relative intensities were determined by visual comparison of the reflections with the intensity scale described in Part I. Values of  $(\sin \theta)/\lambda$  were computed graphically rather than from film measurements. The Lorentz and Polarization and the Tunell factor where applicable were applied to the intensities to obtain relative  $F^2$  values. By careful cross-calibration all data were reduced to the four hour zero layer  $a_1$  axis photograph. Excellent checks were obtained for reflections common to two or more films. The relative  $F^2$  values are listed in Table III.







TABLE III

Rel.			Rel.		
$hkl$	$\frac{\sin \theta}{\lambda}$	$ F_{hkl} ^2$	$hkl$	$\frac{\sin \theta}{\lambda}$	$ F_{hkl} ^2$
010	0.089	950	111	0.201	280
020	0.178	800	121	0.272	1.8
030	0.255	660	131	0.357	1750
040	0.355	24	141	0.435	13
050	0.443	176	151	0.526	0
060	0.533	2.4	161	0.610	-
070	0.620	0	10 $\bar{1}$	0.084	1300
100	0.084	1600	11 $\bar{1}$	0.136	150
110	0.156	370	12 $\bar{1}$	0.208	5800
120	0.234	40	13 $\bar{1}$	0.292	52
130	0.311	72	14 $\bar{1}$	0.376	110
140	0.402	100	15 $\bar{1}$	0.467	111
150	0.486	33	16 $\bar{1}$	0.551	0
160	0.578	-	17 $\bar{1}$	0.636	0
1 $\bar{1}$ 0	0.097	2000	1 $\bar{1}$ 1	0.123	85
1 $\bar{2}$ 0	0.162	100	1 $\bar{2}$ 1	0.188	94
1 $\bar{3}$ 0	0.240	1000	1 $\bar{3}$ 1	0.272	630
1 $\bar{4}$ 0	0.331	1750	1 $\bar{4}$ 1	0.357	5
1 $\bar{5}$ 0	0.415	3.6	1 $\bar{5}$ 1	0.441	159
1 $\bar{6}$ 0	0.565	35	1 $\bar{6}$ 1	0.532	110
1 $\bar{7}$ 0	0.636	1.2	1 $\bar{1}$ $\bar{1}$	0.130	62
001	0.077	660	1 $\bar{2}$ $\bar{1}$	0.149	980
011	0.136	625	1 $\bar{3}$ $\bar{1}$	0.234	610
021	0.218	140	1 $\bar{4}$ $\bar{1}$	0.318	230
031	0.300	145	1 $\bar{5}$ $\bar{1}$	0.396	410
041	0.388	14	1 $\bar{6}$ $\bar{1}$	0.480	195
051	0.475	12	1 $\bar{7}$ $\bar{1}$	0.571	3
061	0.564	2	002	0.154	337
01 $\bar{1}$	0.094	2300	012	0.204	5595
02 $\bar{1}$	0.166	350	022	0.274	580
03 $\bar{1}$	0.248	60	032	0.351	22
04 $\bar{1}$	0.334	16	042	0.434	64
05 $\bar{1}$	0.422	62	052	0.518	37
06 $\bar{1}$	0.511	2.7	062	0.604	6
07 $\bar{1}$	0.597	0.7	01 $\bar{2}$	0.147	155
101	0.143	860	02 $\bar{2}$	0.189	900

# TABLE III

Left			Right		
$\lambda$	$\mu$	$\nu$	$\lambda$	$\mu$	$\nu$
0.00	0.000	0.000	0.00	0.000	0.000
0.01	0.000	0.000	0.01	0.000	0.000
0.02	0.000	0.000	0.02	0.000	0.000
0.03	0.000	0.000	0.03	0.000	0.000
0.04	0.000	0.000	0.04	0.000	0.000
0.05	0.000	0.000	0.05	0.000	0.000
0.06	0.000	0.000	0.06	0.000	0.000
0.07	0.000	0.000	0.07	0.000	0.000
0.08	0.000	0.000	0.08	0.000	0.000
0.09	0.000	0.000	0.09	0.000	0.000
0.10	0.000	0.000	0.10	0.000	0.000
0.11	0.000	0.000	0.11	0.000	0.000
0.12	0.000	0.000	0.12	0.000	0.000
0.13	0.000	0.000	0.13	0.000	0.000
0.14	0.000	0.000	0.14	0.000	0.000
0.15	0.000	0.000	0.15	0.000	0.000
0.16	0.000	0.000	0.16	0.000	0.000
0.17	0.000	0.000	0.17	0.000	0.000
0.18	0.000	0.000	0.18	0.000	0.000
0.19	0.000	0.000	0.19	0.000	0.000
0.20	0.000	0.000	0.20	0.000	0.000
0.21	0.000	0.000	0.21	0.000	0.000
0.22	0.000	0.000	0.22	0.000	0.000
0.23	0.000	0.000	0.23	0.000	0.000
0.24	0.000	0.000	0.24	0.000	0.000
0.25	0.000	0.000	0.25	0.000	0.000
0.26	0.000	0.000	0.26	0.000	0.000
0.27	0.000	0.000	0.27	0.000	0.000
0.28	0.000	0.000	0.28	0.000	0.000
0.29	0.000	0.000	0.29	0.000	0.000
0.30	0.000	0.000	0.30	0.000	0.000
0.31	0.000	0.000	0.31	0.000	0.000
0.32	0.000	0.000	0.32	0.000	0.000
0.33	0.000	0.000	0.33	0.000	0.000
0.34	0.000	0.000	0.34	0.000	0.000
0.35	0.000	0.000	0.35	0.000	0.000
0.36	0.000	0.000	0.36	0.000	0.000
0.37	0.000	0.000	0.37	0.000	0.000
0.38	0.000	0.000	0.38	0.000	0.000
0.39	0.000	0.000	0.39	0.000	0.000
0.40	0.000	0.000	0.40	0.000	0.000
0.41	0.000	0.000	0.41	0.000	0.000
0.42	0.000	0.000	0.42	0.000	0.000
0.43	0.000	0.000	0.43	0.000	0.000
0.44	0.000	0.000	0.44	0.000	0.000
0.45	0.000	0.000	0.45	0.000	0.000
0.46	0.000	0.000	0.46	0.000	0.000
0.47	0.000	0.000	0.47	0.000	0.000
0.48	0.000	0.000	0.48	0.000	0.000
0.49	0.000	0.000	0.49	0.000	0.000
0.50	0.000	0.000	0.50	0.000	0.000

TABLE III (Continued)

Rel.			Rel.		
$hkl$	$\frac{\sin \theta}{\lambda}$	$ F_{hkl} ^2$	$hkl$	$\frac{\sin \theta}{\lambda}$	$ F_{hkl} ^2$
03 $\bar{2}$	0.254	15	043	0.485	37
04 $\bar{2}$	0.332	613	053	0.565	0
05 $\bar{2}$	0.414	14.7	063	0.645	0
06 $\bar{2}$	0.495	39			
07 $\bar{2}$	0.584	22	01 $\bar{3}$	0.214	370
			02 $\bar{3}$	0.235	8
102	0.208	430	03 $\bar{3}$	0.282	19
112	0.259	1000	04 $\bar{3}$	0.347	340
122	0.331	6	05 $\bar{3}$	0.421	96
132	0.402	2	06 $\bar{3}$	0.498	40
142	0.486	47	07 $\bar{3}$	0.580	0
152	0.571	0			
			103	0.379	22
10 $\bar{2}$	0.149	330	113	0.321	25
11 $\bar{2}$	0.162	7.5	123	0.389	10
12 $\bar{2}$	0.214	5	133	0.461	52
13 $\bar{2}$	0.285	600	143	0.538	75
14 $\bar{2}$	0.370	270	153	0.545	0
15 $\bar{2}$	0.454	0			
16 $\bar{2}$	0.538	-	10 $\bar{3}$	0.214	108
17 $\bar{2}$	0.623	-	11 $\bar{3}$	0.214	6970
			12 $\bar{3}$	0.246	53
112	0.175	190	13 $\bar{3}$	0.305	360
122	0.240	57	14 $\bar{3}$	0.376	370
132	0.318	19	15 $\bar{3}$	0.454	26
142	0.396	85	16 $\bar{3}$	0.538	-
152	0.480	218	17 $\bar{3}$	0.590	-
162	0.565	37			
			113	0.246	210
11 $\bar{2}$	0.188	510	123	0.305	70
12 $\bar{2}$	0.208	900	133	0.370	50
13 $\bar{2}$	0.252	2200	143	0.447	80
14 $\bar{2}$	0.324	2.9	153	0.532	13
15 $\bar{2}$	0.396	17	163	0.610	15
16 $\bar{2}$	0.473	112			
17 $\bar{2}$	0.519	6.4	11 $\bar{3}$	0.253	960
			12 $\bar{3}$	0.260	1100
003	0.230	590	13 $\bar{3}$	0.292	180
013	0.275	760	14 $\bar{3}$	0.344	180
023	0.338	72	15 $\bar{3}$	0.409	285
033	0.407	10.8	16 $\bar{3}$	0.480	16



TABLE III (Continued)

Wavelength (microns)	Refractive Index	Dispersion	Wavelength (microns)	Refractive Index	Dispersion
0.35	0.400	0.000	0.35	0.400	0.000
0.40	0.395	0.000	0.40	0.395	0.000
0.45	0.390	0.000	0.45	0.390	0.000
0.50	0.385	0.000	0.50	0.385	0.000
0.55	0.380	0.000	0.55	0.380	0.000
0.60	0.375	0.000	0.60	0.375	0.000
0.65	0.370	0.000	0.65	0.370	0.000
0.70	0.365	0.000	0.70	0.365	0.000
0.75	0.360	0.000	0.75	0.360	0.000
0.80	0.355	0.000	0.80	0.355	0.000
0.85	0.350	0.000	0.85	0.350	0.000
0.90	0.345	0.000	0.90	0.345	0.000
0.95	0.340	0.000	0.95	0.340	0.000
1.00	0.335	0.000	1.00	0.335	0.000
1.05	0.330	0.000	1.05	0.330	0.000
1.10	0.325	0.000	1.10	0.325	0.000
1.15	0.320	0.000	1.15	0.320	0.000
1.20	0.315	0.000	1.20	0.315	0.000
1.25	0.310	0.000	1.25	0.310	0.000
1.30	0.305	0.000	1.30	0.305	0.000
1.35	0.300	0.000	1.35	0.300	0.000
1.40	0.295	0.000	1.40	0.295	0.000
1.45	0.290	0.000	1.45	0.290	0.000
1.50	0.285	0.000	1.50	0.285	0.000
1.55	0.280	0.000	1.55	0.280	0.000
1.60	0.275	0.000	1.60	0.275	0.000
1.65	0.270	0.000	1.65	0.270	0.000
1.70	0.265	0.000	1.70	0.265	0.000
1.75	0.260	0.000	1.75	0.260	0.000
1.80	0.255	0.000	1.80	0.255	0.000
1.85	0.250	0.000	1.85	0.250	0.000
1.90	0.245	0.000	1.90	0.245	0.000
1.95	0.240	0.000	1.95	0.240	0.000
2.00	0.235	0.000	2.00	0.235	0.000
2.05	0.230	0.000	2.05	0.230	0.000
2.10	0.225	0.000	2.10	0.225	0.000
2.15	0.220	0.000	2.15	0.220	0.000
2.20	0.215	0.000	2.20	0.215	0.000
2.25	0.210	0.000	2.25	0.210	0.000
2.30	0.205	0.000	2.30	0.205	0.000
2.35	0.200	0.000	2.35	0.200	0.000
2.40	0.195	0.000	2.40	0.195	0.000
2.45	0.190	0.000	2.45	0.190	0.000
2.50	0.185	0.000	2.50	0.185	0.000
2.55	0.180	0.000	2.55	0.180	0.000
2.60	0.175	0.000	2.60	0.175	0.000
2.65	0.170	0.000	2.65	0.170	0.000
2.70	0.165	0.000	2.70	0.165	0.000
2.75	0.160	0.000	2.75	0.160	0.000
2.80	0.155	0.000	2.80	0.155	0.000
2.85	0.150	0.000	2.85	0.150	0.000
2.90	0.145	0.000	2.90	0.145	0.000
2.95	0.140	0.000	2.95	0.140	0.000
3.00	0.135	0.000	3.00	0.135	0.000
3.05	0.130	0.000	3.05	0.130	0.000
3.10	0.125	0.000	3.10	0.125	0.000
3.15	0.120	0.000	3.15	0.120	0.000
3.20	0.115	0.000	3.20	0.115	0.000
3.25	0.110	0.000	3.25	0.110	0.000
3.30	0.105	0.000	3.30	0.105	0.000
3.35	0.100	0.000	3.35	0.100	0.000
3.40	0.095	0.000	3.40	0.095	0.000
3.45	0.090	0.000	3.45	0.090	0.000
3.50	0.085	0.000	3.50	0.085	0.000
3.55	0.080	0.000	3.55	0.080	0.000
3.60	0.075	0.000	3.60	0.075	0.000
3.65	0.070	0.000	3.65	0.070	0.000
3.70	0.065	0.000	3.70	0.065	0.000
3.75	0.060	0.000	3.75	0.060	0.000
3.80	0.055	0.000	3.80	0.055	0.000
3.85	0.050	0.000	3.85	0.050	0.000
3.90	0.045	0.000	3.90	0.045	0.000
3.95	0.040	0.000	3.95	0.040	0.000
4.00	0.035	0.000	4.00	0.035	0.000



TABLE III (Continued)

Rel.			Rel.		
$hkl$	$\frac{\sin \theta}{\lambda}$	$ F_{hkl} ^2$	$hkl$	$\frac{\sin \theta}{\lambda}$	$ F_{hkl} ^2$
I73	0.558	2.5	I44	0.383	120
004	0.307	25	I54	0.435	640
014	0.350	24	I64	0.499	16
024	0.405	620	I74	0.571	0
034	0.472	0	005	0.384	5.4
044	0.540	0	015	0.423	0
054	0.622	0	025	0.477	0
014	0.287	610	035	0.533	6.4
024	0.296	48	045	0.607	0
034	0.327	800	015	0.363	86
044	0.377	430	025	0.361	0
054	0.441	43	035	0.381	67
064	0.513	175	045	0.420	110
074	0.587	0	055	0.473	6
104	0.357	8	065	0.535	0
114	0.402	15	075	0.604	14.5
124	0.461	7	105	0.428	139
134	0.532	0	115	0.474	24
144	0.596	5	125	0.499	5
104	0.285	240	135	0.590	10
114	0.279	24	105	0.357	2300
124	0.298	900	115	0.344	2.2
134	0.337	415	125	0.331	200
144	0.396	18	135	0.382	370
154	0.461	88	145	0.428	4.6
164	0.538	-	155	0.486	0
174	0.616	-	165	0.558	-
I14	0.318	3	175	0.629	-
I24	0.369	21	I15	0.389	3.5
I34	0.434	63	I25	0.441	33
I44	0.506	0	I35	0.499	6
I54	0.584	0	I45	0.571	19
I14	0.331	73	I55	0.636	6.4
I24	0.324	1200	I15	0.402	185
I34	0.344	10	I25	0.389	8

TABLE III (Continued)

Left			Right		
$\lambda$	$\frac{S}{\lambda}$	$\lambda$	$\lambda$	$\frac{S}{\lambda}$	$\lambda$
1.00	0.000	1.00	1.00	0.000	1.00
1.01	0.000	1.01	1.01	0.000	1.01
1.02	0.000	1.02	1.02	0.000	1.02
1.03	0.000	1.03	1.03	0.000	1.03
1.04	0.000	1.04	1.04	0.000	1.04
1.05	0.000	1.05	1.05	0.000	1.05
1.06	0.000	1.06	1.06	0.000	1.06
1.07	0.000	1.07	1.07	0.000	1.07
1.08	0.000	1.08	1.08	0.000	1.08
1.09	0.000	1.09	1.09	0.000	1.09
1.10	0.000	1.10	1.10	0.000	1.10
1.11	0.000	1.11	1.11	0.000	1.11
1.12	0.000	1.12	1.12	0.000	1.12
1.13	0.000	1.13	1.13	0.000	1.13
1.14	0.000	1.14	1.14	0.000	1.14
1.15	0.000	1.15	1.15	0.000	1.15
1.16	0.000	1.16	1.16	0.000	1.16
1.17	0.000	1.17	1.17	0.000	1.17
1.18	0.000	1.18	1.18	0.000	1.18
1.19	0.000	1.19	1.19	0.000	1.19
1.20	0.000	1.20	1.20	0.000	1.20
1.21	0.000	1.21	1.21	0.000	1.21
1.22	0.000	1.22	1.22	0.000	1.22
1.23	0.000	1.23	1.23	0.000	1.23
1.24	0.000	1.24	1.24	0.000	1.24
1.25	0.000	1.25	1.25	0.000	1.25
1.26	0.000	1.26	1.26	0.000	1.26
1.27	0.000	1.27	1.27	0.000	1.27
1.28	0.000	1.28	1.28	0.000	1.28
1.29	0.000	1.29	1.29	0.000	1.29
1.30	0.000	1.30	1.30	0.000	1.30
1.31	0.000	1.31	1.31	0.000	1.31
1.32	0.000	1.32	1.32	0.000	1.32
1.33	0.000	1.33	1.33	0.000	1.33
1.34	0.000	1.34	1.34	0.000	1.34
1.35	0.000	1.35	1.35	0.000	1.35
1.36	0.000	1.36	1.36	0.000	1.36
1.37	0.000	1.37	1.37	0.000	1.37
1.38	0.000	1.38	1.38	0.000	1.38
1.39	0.000	1.39	1.39	0.000	1.39
1.40	0.000	1.40	1.40	0.000	1.40
1.41	0.000	1.41	1.41	0.000	1.41
1.42	0.000	1.42	1.42	0.000	1.42
1.43	0.000	1.43	1.43	0.000	1.43
1.44	0.000	1.44	1.44	0.000	1.44
1.45	0.000	1.45	1.45	0.000	1.45
1.46	0.000	1.46	1.46	0.000	1.46
1.47	0.000	1.47	1.47	0.000	1.47
1.48	0.000	1.48	1.48	0.000	1.48
1.49	0.000	1.49	1.49	0.000	1.49
1.50	0.000	1.50	1.50	0.000	1.50

TABLE III (Continued)

Rel.			Rel.		
$hkl$	$\frac{\sin \theta}{\lambda}$	$(F_{hkl})^2$	$hkl$	$\frac{\sin \theta}{\lambda}$	$(F_{hkl})^2$
I35	0.402	300	I66	0.571	0
I45	0.428	3.1	I76	0.623	0
I55	0.474	20	007	0.539	14
I65	0.526	3	017	0.570	11
I75	0.590	0	027	0.623	0
006	0.462	110	017	0.514	0
016	0.500	3	027	0.504	6
026	0.549	0	037	0.510	0
036	0.608	30	047	0.530	0
016	0.438	7.3	057	0.563	0
026	0.432	260	067	0.608	10.8
036	0.443	180	107	0.584	4
046	0.472	7.5	117	0.623	0
056	0.516	193	107	0.513	10
066	0.569	0	117	0.493	40
076	0.630	0	127	0.487	115
106	0.506	5	137	0.499	0
116	0.552	0	147	0.526	0
126	0.603	8.5	157	0.571	5.3
106	0.434	14	167	0.623	-
116	0.415	270	I17	0.545	105
126	0.422	40	I27	0.590	6
136	0.441	0	I37	0.643	1.1
146	0.474	80	I17	0.558	12
156	0.525	9	I27	0.538	110
166	0.590	-	I37	0.538	0
I16	0.467	125	I47	0.551	0
I26	0.519	4	I57	0.577	0
I36	0.571	1.6	I67	0.616	0
I46	0.640	3	008	0.616	0
I16	0.480	140	018	0.591	4
I26	0.467	18.5	028	0.578	0
I36	0.467	140	038	0.578	5.3
I46	0.486	180			
I56	0.519	0			



TABLE II  
(Continued)

Year	1961	1962	1963	1964	1965	1966	1967	1968	1969	1970	1971	1972	1973	1974	1975	1976	1977	1978	1979	1980	1981	1982	1983	1984	1985	1986	1987	1988	1989	1990	1991	1992	1993	1994	1995	1996	1997	1998	1999	2000	2001	2002	2003	2004	2005	2006	2007	2008	2009	2010	2011	2012	2013	2014	2015	2016	2017	2018	2019	2020	2021	2022	2023	2024	2025	2026	2027	2028	2029	2030	2031	2032	2033	2034	2035	2036	2037	2038	2039	2040	2041	2042	2043	2044	2045	2046	2047	2048	2049	2050	2051	2052	2053	2054	2055	2056	2057	2058	2059	2060	2061	2062	2063	2064	2065	2066	2067	2068	2069	2070	2071	2072	2073	2074	2075	2076	2077	2078	2079	2080	2081	2082	2083	2084	2085	2086	2087	2088	2089	2090	2091	2092	2093	2094	2095	2096	2097	2098	2099	2100
1961	1962	1963	1964	1965	1966	1967	1968	1969	1970	1971	1972	1973	1974	1975	1976	1977	1978	1979	1980	1981	1982	1983	1984	1985	1986	1987	1988	1989	1990	1991	1992	1993	1994	1995	1996	1997	1998	1999	2000	2001	2002	2003	2004	2005	2006	2007	2008	2009	2010	2011	2012	2013	2014	2015	2016	2017	2018	2019	2020	2021	2022	2023	2024	2025	2026	2027	2028	2029	2030	2031	2032	2033	2034	2035	2036	2037	2038	2039	2040	2041	2042	2043	2044	2045	2046	2047	2048	2049	2050	2051	2052	2053	2054	2055	2056	2057	2058	2059	2060	2061	2062	2063	2064	2065	2066	2067	2068	2069	2070	2071	2072	2073	2074	2075	2076	2077	2078	2079	2080	2081	2082	2083	2084	2085	2086	2087	2088	2089	2090	2091	2092	2093	2094	2095	2096	2097	2098	2099	2100	



TABLE III (Continued)

Rel.			Rel.		
hkl	$\frac{\sin \theta}{\lambda}$	$ F_{hkl} ^2$	hkl	$\frac{\sin \theta}{\lambda}$	$ F_{hkl} ^2$
04 $\bar{8}$	0.592	50	201	0.227	400
05 $\bar{8}$	0.620	0	211	0.285	485
10 $\bar{3}$	0.597	14	221	0.350	42
11 $\bar{8}$	0.571	0	231	0.422	95
12 $\bar{8}$	0.564	1.6	241	0.499	17
13 $\bar{8}$	0.564	10.4	251	0.583	0
14 $\bar{8}$	0.584	0	20 $\bar{1}$	0.175	880
15 $\bar{8}$	0.622	0	21 $\bar{1}$	0.214	14
11 $\bar{8}$	0.624	4	22 $\bar{1}$	0.278	200
11 $\bar{3}$	0.629	6.1	23 $\bar{1}$	0.357	120
12 $\bar{8}$	0.616	0	24 $\bar{1}$	0.441	0
13 $\bar{8}$	0.603	2.6	25 $\bar{1}$	0.519	0
14 $\bar{8}$	0.616	0	26 $\bar{1}$	0.603	-
15 $\bar{8}$	0.636	0	211	0.169	314
02 $\bar{9}$	0.647	-	221	0.208	6550
03 $\bar{9}$	0.645	0	231	0.272	108
11 $\bar{9}$	0.640	14.7	241	0.350	17
12 $\bar{9}$	0.624	-	251	0.428	290
13 $\bar{9}$	0.624	-	261	0.512	1.7
14 $\bar{9}$	0.635	-	271	0.585	0
200	0.188	600	21 $\bar{1}$	0.208	4100
210	0.240	1180	22 $\bar{1}$	0.214	80
220	0.305	200	23 $\bar{1}$	0.259	70
230	0.383	300	24 $\bar{1}$	0.324	700
240	0.467	0	25 $\bar{1}$	0.396	0
250	0.545	0	26 $\bar{1}$	0.473	160
260	0.636	0	271	0.558	20
210	0.175	27.5	202	0.285	167
220	0.194	25.	212	0.337	13
230	0.259	500	222	0.402	71
240	0.324	130	232	0.473	52
250	0.402	15	242	0.551	17
260	0.487	120	252	0.629	0
270	0.571	1.8	20 $\bar{2}$	0.188	800
			21 $\bar{2}$	0.214	230
			22 $\bar{2}$	0.272	840

## TABLE 2.1 (Continued)

Year	Value	Year	Value	Year
(1960)	(1960)	(1960)	(1960)	(1960)
1960	100.0	1960	100.0	1960
1961	101.0	1961	101.0	1961
1962	102.0	1962	102.0	1962
1963	103.0	1963	103.0	1963
1964	104.0	1964	104.0	1964
1965	105.0	1965	105.0	1965
1966	106.0	1966	106.0	1966
1967	107.0	1967	107.0	1967
1968	108.0	1968	108.0	1968
1969	109.0	1969	109.0	1969
1970	110.0	1970	110.0	1970
1971	111.0	1971	111.0	1971
1972	112.0	1972	112.0	1972
1973	113.0	1973	113.0	1973
1974	114.0	1974	114.0	1974
1975	115.0	1975	115.0	1975
1976	116.0	1976	116.0	1976
1977	117.0	1977	117.0	1977
1978	118.0	1978	118.0	1978
1979	119.0	1979	119.0	1979
1980	120.0	1980	120.0	1980
1981	121.0	1981	121.0	1981
1982	122.0	1982	122.0	1982
1983	123.0	1983	123.0	1983
1984	124.0	1984	124.0	1984
1985	125.0	1985	125.0	1985
1986	126.0	1986	126.0	1986
1987	127.0	1987	127.0	1987
1988	128.0	1988	128.0	1988
1989	129.0	1989	129.0	1989
1990	130.0	1990	130.0	1990
1991	131.0	1991	131.0	1991
1992	132.0	1992	132.0	1992
1993	133.0	1993	133.0	1993
1994	134.0	1994	134.0	1994
1995	135.0	1995	135.0	1995
1996	136.0	1996	136.0	1996
1997	137.0	1997	137.0	1997
1998	138.0	1998	138.0	1998
1999	139.0	1999	139.0	1999
2000	140.0	2000	140.0	2000
2001	141.0	2001	141.0	2001
2002	142.0	2002	142.0	2002
2003	143.0	2003	143.0	2003
2004	144.0	2004	144.0	2004
2005	145.0	2005	145.0	2005
2006	146.0	2006	146.0	2006
2007	147.0	2007	147.0	2007
2008	148.0	2008	148.0	2008
2009	149.0	2009	149.0	2009
2010	150.0	2010	150.0	2010
2011	151.0	2011	151.0	2011
2012	152.0	2012	152.0	2012
2013	153.0	2013	153.0	2013
2014	154.0	2014	154.0	2014
2015	155.0	2015	155.0	2015
2016	156.0	2016	156.0	2016
2017	157.0	2017	157.0	2017
2018	158.0	2018	158.0	2018
2019	159.0	2019	159.0	2019
2020	160.0	2020	160.0	2020
2021	161.0	2021	161.0	2021
2022	162.0	2022	162.0	2022
2023	163.0	2023	163.0	2023
2024	164.0	2024	164.0	2024
2025	165.0	2025	165.0	2025
2026	166.0	2026	166.0	2026
2027	167.0	2027	167.0	2027
2028	168.0	2028	168.0	2028
2029	169.0	2029	169.0	2029
2030	170.0	2030	170.0	2030
2031	171.0	2031	171.0	2031
2032	172.0	2032	172.0	2032
2033	173.0	2033	173.0	2033
2034	174.0	2034	174.0	2034
2035	175.0	2035	175.0	2035
2036	176.0	2036	176.0	2036
2037	177.0	2037	177.0	2037
2038	178.0	2038	178.0	2038
2039	179.0	2039	179.0	2039
2040	180.0	2040	180.0	2040
2041	181.0	2041	181.0	2041
2042	182.0	2042	182.0	2042
2043	183.0	2043	183.0	2043
2044	184.0	2044	184.0	2044
2045	185.0	2045	185.0	2045
2046	186.0	2046	186.0	2046
2047	187.0	2047	187.0	2047
2048	188.0	2048	188.0	2048
2049	189.0	2049	189.0	2049
2050	190.0	2050	190.0	2050
2051	191.0	2051	191.0	2051
2052	192.0	2052	192.0	2052
2053	193.0	2053	193.0	2053
2054	194.0	2054	194.0	2054
2055	195.0	2055	195.0	2055
2056	196.0	2056	196.0	2056
2057	197.0	2057	197.0	2057
2058	198.0	2058	198.0	2058
2059	199.0	2059	199.0	2059
2060	200.0	2060	200.0	2060
2061	201.0	2061	201.0	2061
2062	202.0	2062	202.0	2062
2063	203.0	2063	203.0	2063
2064	204.0	2064	204.0	2064
2065	205.0	2065	205.0	2065
2066	206.0	2066	206.0	2066
2067	207.0	2067	207.0	2067
2068	208.0	2068	208.0	2068
2069	209.0	2069	209.0	2069
2070	210.0	2070	210.0	2070
2071	211.0	2071	211.0	2071
2072	212.0	2072	212.0	2072
2073	213.0	2073	213.0	2073
2074	214.0	2074	214.0	2074
2075	215.0	2075	215.0	2075
2076	216.0	2076	216.0	2076
2077	217.0	2077	217.0	2077
2078	218.0	2078	218.0	2078
2079	219.0	2079	219.0	2079
2080	220.0	2080	220.0	2080
2081	221.0	2081	221.0	2081
2082	222.0	2082	222.0	2082
2083	223.0	2083	223.0	2083
2084	224.0	2084	224.0	2084
2085	225.0	2085	225.0	2085
2086	226.0	2086	226.0	2086
2087	227.0	2087	227.0	2087
2088	228.0	2088	228.0	2088
2089	229.0	2089	229.0	2089
2090	230.0	2090	230.0	2090
2091	231.0	2091	231.0	2091
2092	232.0	2092	232.0	2092
2093	233.0	2093	233.0	2093
2094	234.0	2094	234.0	2094
2095	235.0	2095	235.0	2095
2096	236.0	2096	236.0	2096
2097	237.0	2097	237.0	2097
2098	238.0	2098	238.0	2098
2099	239.0	2099	239.0	2099
2100	240.0	2100	240.0	2100

TABLE III (Continued)

hkℓ	Rel.		hkℓ	Rel.	
	$\frac{\sin \theta}{\lambda}$	$ F_{hkl} ^2$		$\frac{\sin \theta}{\lambda}$	$ F_{hkl} ^2$
$\overline{2}3\overline{2}$	0.344	6	$\overline{2}53$	0.506	0
$\overline{2}4\overline{2}$	0.422	260	$\overline{2}63$	0.583	6.6
$\overline{2}5\overline{2}$	0.506	35	$\overline{2}1\overline{3}$	0.318	6.5
$\overline{2}6\overline{2}$	0.584	-	$\overline{2}2\overline{3}$	0.311	9
$\overline{2}1\overline{2}$	0.201	470	$\overline{2}3\overline{3}$	0.331	420
$\overline{2}2\overline{2}$	0.246	15	$\overline{2}4\overline{3}$	0.369	100
$\overline{2}3\overline{2}$	0.305	170	$\overline{2}5\overline{3}$	0.421	500
$\overline{2}4\overline{2}$	0.383	125	$\overline{2}6\overline{3}$	0.487	60
$\overline{2}5\overline{2}$	0.461	61	$\overline{2}7\overline{3}$	0.558	1.4
$\overline{2}6\overline{2}$	0.543	20	$\overline{2}8\overline{3}$	0.630	63
$\overline{2}7\overline{2}$	0.618	0	$\overline{2}04$	0.422	0
$\overline{2}1\overline{2}$	0.260	11	$\overline{2}14$	0.467	80
$\overline{2}2\overline{2}$	0.260	490	$\overline{2}24$	0.499	0
$\overline{2}3\overline{2}$	0.292	150	$\overline{2}34$	0.590	8.8
$\overline{2}4\overline{2}$	0.337	1.4	$\overline{2}0\overline{4}$	0.292	145
$\overline{2}5\overline{2}$	0.402	85	$\overline{2}1\overline{4}$	0.298	400
$\overline{2}6\overline{2}$	0.474	0	$\overline{2}2\overline{4}$	0.324	5.3
$\overline{2}7\overline{2}$	0.551	0	$\overline{2}3\overline{4}$	0.369	900
$\overline{2}8\overline{2}$	0.630	5.5	$\overline{2}4\overline{4}$	0.434	180
$\overline{2}03$	0.350	1500	$\overline{2}5\overline{4}$	0.506	0
$\overline{2}13$	0.402	16	$\overline{2}6\overline{4}$	0.577	26.6
$\overline{2}23$	0.461	80	$\overline{2}14$	0.311	90
$\overline{2}33$	0.532	5	$\overline{2}24$	0.357	1.4
$\overline{2}43$	0.603	13	$\overline{2}34$	0.415	10
$\overline{2}0\overline{3}$	0.235	200	$\overline{2}44$	0.486	70
$\overline{2}1\overline{3}$	0.246	74	$\overline{2}54$	0.558	15
$\overline{2}2\overline{3}$	0.292	300	$\overline{2}64$	0.630	5
$\overline{2}3\overline{3}$	0.344	13	$\overline{2}1\overline{4}$	0.389	110
$\overline{2}4\overline{3}$	0.422	1.9	$\overline{2}2\overline{4}$	0.376	2.3
$\overline{2}5\overline{3}$	0.499	177	$\overline{2}3\overline{4}$	0.389	190
$\overline{2}6\overline{3}$	0.577	0	$\overline{2}4\overline{4}$	0.415	2.8
$\overline{2}13$	0.253	1100	$\overline{2}5\overline{4}$	0.454	113
$\overline{2}23$	0.298	240	$\overline{2}6\overline{4}$	0.512	36
$\overline{2}33$	0.357	1900	$\overline{2}74$	0.572	21
$\overline{2}43$	0.428	34			



[illegible]



TABLE III (Continued)

Rel.			Rel.		
$hkl$	$\frac{\sin \theta}{\lambda}$	$ F_{hkl} ^2$	$hkl$	$\frac{\sin \theta}{\lambda}$	$ F_{hkl} ^2$
205	0.493	27	$\bar{2}1\bar{6}$	0.545	16
215	0.538	51	$\bar{2}2\bar{6}$	0.512	94
225	0.596	2	$\bar{2}3\bar{6}$	0.506	9.2
			$\bar{2}4\bar{6}$	0.525	100
$20\bar{5}$	0.357	2.4	$\bar{2}5\bar{6}$	0.551	1.4
$21\bar{5}$	0.350	184	$\bar{2}6\bar{6}$	0.584	17
$22\bar{5}$	0.369	120	$\bar{2}7\bar{6}$	0.635	4.6
$23\bar{5}$	0.408	50			
$24\bar{5}$	0.461	630	207	0.635	0
$25\bar{5}$	0.519	0			
$26\bar{5}$	0.590	12.6	$20\bar{7}$	0.499	26
			$21\bar{7}$	0.486	68
$\bar{2}15$	0.383	18	$22\bar{7}$	0.486	40
$\bar{2}25$	0.428	115	$23\bar{7}$	0.506	245
$\bar{2}35$	0.480	74	$24\bar{7}$	0.533	0
$\bar{2}45$	0.545	100	$25\bar{7}$	0.583	6.4
$\bar{2}55$	0.616	3	$26\bar{7}$	0.636	-
$\bar{2}1\bar{5}$	0.461	0	$\bar{2}17$	0.525	14.6
$\bar{2}2\bar{5}$	0.447	150	$\bar{2}27$	0.564	0
$\bar{2}3\bar{5}$	0.447	72	$\bar{2}37$	0.617	0
$\bar{2}4\bar{5}$	0.467	118			
$\bar{2}5\bar{5}$	0.499	2.7	$\bar{2}1\bar{7}$	0.610	10
$\bar{2}6\bar{5}$	0.545	0	$\bar{2}2\bar{7}$	0.590	0
$\bar{2}7\bar{5}$	0.596	64	$\bar{2}3\bar{7}$	0.584	0
			$\bar{2}4\bar{7}$	0.584	12
206	0.564	7	$\bar{2}5\bar{7}$	0.603	0
216	0.609	2.9	$\bar{2}6\bar{7}$	0.636	12
$20\bar{6}$	0.428	29	$20\bar{8}$	0.571	0
$21\bar{6}$	0.415	0	$21\bar{8}$	0.558	47
$22\bar{6}$	0.428	450	$22\bar{8}$	0.558	0
$23\bar{6}$	0.454	53	$23\bar{8}$	0.571	1.8
$24\bar{6}$	0.499	9.1	$24\bar{8}$	0.590	9.0
$25\bar{6}$	0.551	8.4	$25\bar{8}$	0.629	7.6
$26\bar{6}$	0.610	-			
			$\bar{2}18$	0.617	25.6
$\bar{2}16$	0.454	8	$\bar{2}28$	0.636	0
$\bar{2}26$	0.493	0			
$\bar{2}36$	0.538	1.6	$20\bar{9}$	0.649	2.5
$\bar{2}46$	0.610	4	$21\bar{9}$	0.630	0

TABLE III (Continued)

Left			Right		
Angle	sin	sec	Angle	sin	sec
10	0.1736	17.36	70	0.9397	93.97
11	0.1928	19.28	71	0.9455	94.55
12	0.2108	21.08	72	0.9511	95.11
13	0.2279	22.79	73	0.9563	95.63
14	0.2449	24.49	74	0.9612	96.12
15	0.2618	26.18	75	0.9658	96.58
16	0.2784	27.84	76	0.9701	97.01
17	0.2949	29.49	77	0.9741	97.41
18	0.3113	31.13	78	0.9778	97.78
19	0.3276	32.76	79	0.9812	98.12
20	0.3438	34.38	80	0.9843	98.43
21	0.3598	35.98	81	0.9871	98.71
22	0.3757	37.57	82	0.9896	98.96
23	0.3914	39.14	83	0.9919	99.19
24	0.4069	40.69	84	0.9940	99.40
25	0.4222	42.22	85	0.9959	99.59
26	0.4374	43.74	86	0.9976	99.76
27	0.4523	45.23	87	0.9990	99.90
28	0.4671	46.71	88	0.9999	99.99
29	0.4817	48.17	89	0.9999	99.99
30	0.4961	49.61	90	1.0000	100.00
31	0.5103	51.03			
32	0.5244	52.44			
33	0.5383	53.83			
34	0.5521	55.21			
35	0.5657	56.57			
36	0.5791	57.91			
37	0.5923	59.23			
38	0.6054	60.54			
39	0.6183	61.83			
40	0.6311	63.11			
41	0.6437	64.37			
42	0.6561	65.61			
43	0.6683	66.83			
44	0.6804	68.04			
45	0.6923	69.23			
46	0.7041	70.41			
47	0.7157	71.57			
48	0.7271	72.71			
49	0.7383	73.83			
50	0.7493	74.93			
51	0.7601	76.01			
52	0.7707	77.07			
53	0.7811	78.11			
54	0.7913	79.13			
55	0.8013	80.13			
56	0.8111	81.11			
57	0.8207	82.07			
58	0.8301	83.01			
59	0.8393	83.93			
60	0.8483	84.83			
61	0.8571	85.71			
62	0.8657	86.57			
63	0.8741	87.41			
64	0.8823	88.23			
65	0.8903	89.03			
66	0.8981	89.81			
67	0.9057	90.57			
68	0.9131	91.31			
69	0.9203	92.03			
70	0.9273	92.73			
71	0.9341	93.41			
72	0.9407	94.07			
73	0.9471	94.71			
74	0.9533	95.33			
75	0.9593	95.93			
76	0.9651	96.51			
77	0.9707	97.07			
78	0.9761	97.61			
79	0.9813	98.13			
80	0.9863	98.63			
81	0.9911	99.11			
82	0.9957	99.57			
83	0.9999	99.99			
84	0.9999	99.99			
85	0.9999	99.99			
86	0.9999	99.99			
87	0.9999	99.99			
88	0.9999	99.99			
89	0.9999	99.99			
90	0.9999	99.99			

TABLE III (Continued)

Rel.			Rel.		
hk $\ell$	$\frac{\sin \theta}{\lambda}$	$ F_{hk\ell} ^2$	hk $\ell$	$\frac{\sin \theta}{\lambda}$	$ F_{hk\ell} ^2$
22 $\bar{9}$	0.621	21	31 $\bar{1}$	0.292	98
23 $\bar{9}$	0.629	0	32 $\bar{1}$	0.292	800
24 $\bar{9}$	0.637	-	33 $\bar{1}$	0.311	230
			34 $\bar{1}$	0.357	380
300	0.279	169	35 $\bar{1}$	0.422	80
310	0.331	860	36 $\bar{1}$	0.486	70
320	0.389	13	37 $\bar{1}$	0.565	0
330	0.461	98	38 $\bar{1}$	0.630	2
340	0.538	0			
350	0.616	-	302	0.369	110
			312	0.422	53
310	0.259	235	322	0.480	97
320	0.266	162	332	0.545	4.6
330	0.298	26	342	0.617	3.4
340	0.356	580			
350	0.422	30	30 $\bar{2}$	0.266	1600
360	0.493	65	31 $\bar{2}$	0.292	200
370	0.571	35	32 $\bar{2}$	0.350	540
380	0.643	-	33 $\bar{2}$	0.415	24
			34 $\bar{2}$	0.486	64
301	0.318	11	35 $\bar{2}$	0.564	0
311	0.370	9	36 $\bar{2}$	0.636	-
321	0.435	85			
331	0.499	0	312	0.259	52
341	0.570	3.4	322	0.285	150
			332	0.331	120
30 $\bar{1}$	0.266	350	342	0.389	0
31 $\bar{1}$	0.305	230	352	0.467	130
32 $\bar{1}$	0.363	3	362	0.545	0
33 $\bar{1}$	0.435	152	372	0.609	0
34 $\bar{1}$	0.506	13			
35 $\bar{1}$	0.584	28	31 $\bar{2}$	0.337	114
			322	0.331	17.5
311	0.247	37	332	0.344	460
321	0.266	38	342	0.383	270
331	0.305	480	352	0.435	90
341	0.370	120	362	0.493	160
351	0.441	120	372	0.558	4
361	0.512	7.5	382	0.630	14
371	0.596	20			



TABLE III (Continued)

Left			Right		
$\lambda$	$\frac{g}{\lambda}$	$\lambda$	$\lambda$	$\frac{g}{\lambda}$	$\lambda$
10	0.000	100	10	0.000	100
20	0.000	200	20	0.000	200
30	0.000	300	30	0.000	300
40	0.000	400	40	0.000	400
50	0.000	500	50	0.000	500
60	0.000	600	60	0.000	600
70	0.000	700	70	0.000	700
80	0.000	800	80	0.000	800
90	0.000	900	90	0.000	900
100	0.000	1000	100	0.000	1000
110	0.000	1100	110	0.000	1100
120	0.000	1200	120	0.000	1200
130	0.000	1300	130	0.000	1300
140	0.000	1400	140	0.000	1400
150	0.000	1500	150	0.000	1500
160	0.000	1600	160	0.000	1600
170	0.000	1700	170	0.000	1700
180	0.000	1800	180	0.000	1800
190	0.000	1900	190	0.000	1900
200	0.000	2000	200	0.000	2000
210	0.000	2100	210	0.000	2100
220	0.000	2200	220	0.000	2200
230	0.000	2300	230	0.000	2300
240	0.000	2400	240	0.000	2400
250	0.000	2500	250	0.000	2500
260	0.000	2600	260	0.000	2600
270	0.000	2700	270	0.000	2700
280	0.000	2800	280	0.000	2800
290	0.000	2900	290	0.000	2900
300	0.000	3000	300	0.000	3000
310	0.000	3100	310	0.000	3100
320	0.000	3200	320	0.000	3200
330	0.000	3300	330	0.000	3300
340	0.000	3400	340	0.000	3400
350	0.000	3500	350	0.000	3500
360	0.000	3600	360	0.000	3600
370	0.000	3700	370	0.000	3700
380	0.000	3800	380	0.000	3800
390	0.000	3900	390	0.000	3900
400	0.000	4000	400	0.000	4000
410	0.000	4100	410	0.000	4100
420	0.000	4200	420	0.000	4200
430	0.000	4300	430	0.000	4300
440	0.000	4400	440	0.000	4400
450	0.000	4500	450	0.000	4500
460	0.000	4600	460	0.000	4600
470	0.000	4700	470	0.000	4700
480	0.000	4800	480	0.000	4800
490	0.000	4900	490	0.000	4900
500	0.000	5000	500	0.000	5000



TABLE III (Continued)

Rel.			Rel.		
hkl	$\frac{\sin \theta}{\lambda}$	$(F_{hkl})^2$	hkl	$\frac{\sin \theta}{\lambda}$	$(F_{hkl})^2$
303	0.428	34	344	0.486	16
313	0.473	4.5	354	0.558	0
323	0.533	4.5	364	0.629	-
333	0.597	2.7			
$\bar{3}0\bar{3}$	0.285	150	$\bar{3}14$	0.337	1525
$\bar{3}1\bar{3}$	0.311	45	$\bar{3}24$	0.370	4
$\bar{3}2\bar{3}$	0.350	640	$\bar{3}34$	0.421	10
$\bar{3}3\bar{3}$	0.415	30	$\bar{3}44$	0.480	30
$\bar{3}4\bar{3}$	0.487	228	$\bar{3}54$	0.551	1.5
$\bar{3}5\bar{3}$	0.558	26	$\bar{3}64$	0.623	8
$\bar{3}6\bar{3}$	0.629	-			
$\bar{3}1\bar{3}$	0.292	530	$\bar{3}1\bar{4}$	0.461	3.2
$\bar{3}2\bar{3}$	0.324	15	$\bar{3}2\bar{4}$	0.441	125
$\bar{3}3\bar{3}$	0.370	21	$\bar{3}3\bar{4}$	0.441	130
$\bar{3}4\bar{3}$	0.435	48	$\bar{3}4\bar{4}$	0.461	0
$\bar{3}5\bar{3}$	0.506	33	$\bar{3}5\bar{4}$	0.493	140
$\bar{3}6\bar{3}$	0.577	0	$\bar{3}6\bar{4}$	0.538	0
$\bar{3}7\bar{3}$	0.654	0	$\bar{3}7\bar{4}$	0.591	11
$\bar{3}1\bar{3}$	0.396	334	305	0.558	26
$\bar{3}2\bar{3}$	0.383	60	315	0.603	8
$\bar{3}3\bar{3}$	0.389	130			
$\bar{3}4\bar{3}$	0.415	45	305	0.383	10
$\bar{3}5\bar{3}$	0.454	0	315	0.389	300
$\bar{3}6\bar{3}$	0.512	40	325	0.409	36
$\bar{3}7\bar{3}$	0.571	0	335	0.454	558
$\bar{3}8\bar{3}$	0.637	-	345	0.506	114
-			355	0.571	0
304	0.493	36	365	0.636	-
314	0.538	12			
324	0.597	2	315	0.395	2.9
			325	0.435	170
$\bar{3}0\bar{4}$	0.331	200	335	0.480	0
$\bar{3}1\bar{4}$	0.337	800	345	0.538	0
$\bar{3}2\bar{4}$	0.376	128	355	0.598	2.3
$\bar{3}3\bar{4}$	0.428	135			
			$\bar{3}1\bar{5}$	0.532	3.9
			325	0.513	12
			335	0.506	0
			345	0.513	14
			355	0.538	0

Year	Price	Quantity	Year	Price	Quantity
1910	100.0	100	1911	100.0	100
1912	100.0	100	1913	100.0	100
1914	100.0	100	1915	100.0	100
1916	100.0	100	1917	100.0	100
1918	100.0	100	1919	100.0	100
1920	100.0	100	1921	100.0	100
1922	100.0	100	1923	100.0	100
1924	100.0	100	1925	100.0	100
1926	100.0	100	1927	100.0	100
1928	100.0	100	1929	100.0	100
1930	100.0	100	1931	100.0	100
1932	100.0	100	1933	100.0	100
1934	100.0	100	1935	100.0	100
1936	100.0	100	1937	100.0	100
1938	100.0	100	1939	100.0	100
1940	100.0	100	1941	100.0	100
1942	100.0	100	1943	100.0	100
1944	100.0	100	1945	100.0	100
1946	100.0	100	1947	100.0	100
1948	100.0	100	1949	100.0	100
1950	100.0	100	1951	100.0	100
1952	100.0	100	1953	100.0	100
1954	100.0	100	1955	100.0	100
1956	100.0	100	1957	100.0	100
1958	100.0	100	1959	100.0	100
1960	100.0	100	1961	100.0	100
1962	100.0	100	1963	100.0	100
1964	100.0	100	1965	100.0	100
1966	100.0	100	1967	100.0	100
1968	100.0	100	1969	100.0	100
1970	100.0	100	1971	100.0	100
1972	100.0	100	1973	100.0	100
1974	100.0	100	1975	100.0	100
1976	100.0	100	1977	100.0	100
1978	100.0	100	1979	100.0	100
1980	100.0	100	1981	100.0	100
1982	100.0	100	1983	100.0	100
1984	100.0	100	1985	100.0	100
1986	100.0	100	1987	100.0	100
1988	100.0	100	1989	100.0	100
1990	100.0	100	1991	100.0	100
1992	100.0	100	1993	100.0	100
1994	100.0	100	1995	100.0	100
1996	100.0	100	1997	100.0	100
1998	100.0	100	1999	100.0	100
2000	100.0	100	2001	100.0	100
2002	100.0	100	2003	100.0	100
2004	100.0	100	2005	100.0	100
2006	100.0	100	2007	100.0	100
2008	100.0	100	2009	100.0	100
2010	100.0	100	2011	100.0	100
2012	100.0	100	2013	100.0	100
2014	100.0	100	2015	100.0	100
2016	100.0	100	2017	100.0	100
2018	100.0	100	2019	100.0	100
2020	100.0	100	2021	100.0	100
2022	100.0	100	2023	100.0	100
2024	100.0	100	2025	100.0	100

TABLE III (Continued)

Rel.			Rel.		
hkl	$\frac{\sin \theta}{\lambda}$	$ F_{hkl} ^2$	hkl	$\frac{\sin \theta}{\lambda}$	$ F_{hkl} ^2$
$\bar{3}65$	0.577	0	$30\bar{9}$	0.577	0
$\bar{3}75$	0.623	1.8	$31\bar{8}$	0.571	2.4
$306$	0.629	0	$32\bar{8}$	0.571	2.4
$30\bar{6}$	0.441	320	$33\bar{8}$	0.584	0
$31\bar{6}$	0.441	45	$34\bar{8}$	0.616	0
$32\bar{6}$	0.454	51	$\bar{3}18$	0.596	5.4
$33\bar{6}$	0.486	24	$\bar{3}28$	0.630	6.8
$34\bar{6}$	0.532	20	$30\bar{9}$	0.641	1.3
$35\bar{6}$	0.590	9.5	$31\bar{9}$	0.630	4.1
$36\bar{6}$	0.648	-	$32\bar{9}$	0.630	1.3
$\bar{3}16$	0.461	115	$33\bar{9}$	0.641	14
$\bar{3}26$	0.493	280	$400$	0.376	130
$\bar{3}36$	0.538	14.5	$410$	0.422	13
$\bar{3}46$	0.597	0	$420$	0.473	90
$\bar{3}56$	0.650	0.1	$430$	0.538	0
$\bar{3}1\bar{6}$	0.597	6.2	$440$	0.610	1.1
$\bar{3}2\bar{6}$	0.571	2.8	$\bar{4}10$	0.350	300
$\bar{3}3\bar{6}$	0.564	9.4	$\bar{4}20$	0.350	210
$\bar{3}4\bar{6}$	0.571	17	$\bar{4}30$	0.363	400
$\bar{3}5\bar{6}$	0.591	0	$\bar{4}40$	0.402	90
$36\bar{6}$	0.616	10	$\bar{4}50$	0.454	25
$30\bar{7}$	0.506	45	$\bar{4}60$	0.512	0
$31\bar{7}$	0.499	17.5	$\bar{4}70$	0.577	0
$32\bar{7}$	0.512	145	$401$	0.408	75
$33\bar{7}$	0.532	21.8	$411$	0.454	84
$34\bar{7}$	0.571	6.1	$421$	0.519	77
$35\bar{7}$	0.616	14.8	$431$	0.577	0
$\bar{3}17$	0.525	32	$40\bar{1}$	0.357	80
$\bar{3}27$	0.558	16	$41\bar{1}$	0.389	310
$\bar{3}37$	0.610	9.5	$42\bar{1}$	0.447	90
$\bar{3}2\bar{7}$	0.642	-	$43\bar{1}$	0.512	0
$\bar{3}3\bar{7}$	0.629	-	$44\bar{1}$	0.578	4.1
$\bar{3}4\bar{7}$	0.629	-	$\bar{4}11$	0.337	80
$\bar{3}5\bar{7}$	0.642	-	$\bar{4}21$	0.337	500



(Continued) TABLE III

Left			Right		
$\lambda$	$\frac{u}{\lambda}$	$\frac{v}{\lambda}$	$\lambda$	$\frac{u}{\lambda}$	$\frac{v}{\lambda}$
0	0.000	0.000	0	0.000	0.000
0.5	0.000	0.000	0.5	0.000	0.000
1.0	0.000	0.000	1.0	0.000	0.000
1.5	0.000	0.000	1.5	0.000	0.000
2.0	0.000	0.000	2.0	0.000	0.000
2.5	0.000	0.000	2.5	0.000	0.000
3.0	0.000	0.000	3.0	0.000	0.000
3.5	0.000	0.000	3.5	0.000	0.000
4.0	0.000	0.000	4.0	0.000	0.000
4.5	0.000	0.000	4.5	0.000	0.000
5.0	0.000	0.000	5.0	0.000	0.000
5.5	0.000	0.000	5.5	0.000	0.000
6.0	0.000	0.000	6.0	0.000	0.000
6.5	0.000	0.000	6.5	0.000	0.000
7.0	0.000	0.000	7.0	0.000	0.000
7.5	0.000	0.000	7.5	0.000	0.000
8.0	0.000	0.000	8.0	0.000	0.000
8.5	0.000	0.000	8.5	0.000	0.000
9.0	0.000	0.000	9.0	0.000	0.000
9.5	0.000	0.000	9.5	0.000	0.000
10.0	0.000	0.000	10.0	0.000	0.000
10.5	0.000	0.000	10.5	0.000	0.000
11.0	0.000	0.000	11.0	0.000	0.000
11.5	0.000	0.000	11.5	0.000	0.000
12.0	0.000	0.000	12.0	0.000	0.000
12.5	0.000	0.000	12.5	0.000	0.000
13.0	0.000	0.000	13.0	0.000	0.000
13.5	0.000	0.000	13.5	0.000	0.000
14.0	0.000	0.000	14.0	0.000	0.000
14.5	0.000	0.000	14.5	0.000	0.000
15.0	0.000	0.000	15.0	0.000	0.000
15.5	0.000	0.000	15.5	0.000	0.000
16.0	0.000	0.000	16.0	0.000	0.000
16.5	0.000	0.000	16.5	0.000	0.000
17.0	0.000	0.000	17.0	0.000	0.000
17.5	0.000	0.000	17.5	0.000	0.000
18.0	0.000	0.000	18.0	0.000	0.000
18.5	0.000	0.000	18.5	0.000	0.000
19.0	0.000	0.000	19.0	0.000	0.000
19.5	0.000	0.000	19.5	0.000	0.000
20.0	0.000	0.000	20.0	0.000	0.000



TABLE III (Continued)

Rel.			Rel.		
$hkl$	$\frac{\sin \theta}{\lambda}$	$ F_{hkl} ^2$	$hkl$	$\frac{\sin \theta}{\lambda}$	$ F_{hkl} ^2$
$\bar{4}31$	0.363	34	$\bar{4}8\bar{2}$	0.648	-
$\bar{4}41$	0.402	15	403	0.512	9.1
$\bar{4}51$	0.461	130	413	0.558	0
$\bar{4}61$	0.525	0	423	0.596	8.9
$\bar{4}71$	0.596	15	40 $\bar{3}$	0.357	48
41 $\bar{1}$	0.383	115	41 $\bar{3}$	0.383	160
42 $\bar{1}$	0.376	90	42 $\bar{3}$	0.428	123
43 $\bar{1}$	0.383	6	43 $\bar{3}$	0.486	19
44 $\bar{1}$	0.408	190	44 $\bar{3}$	0.551	0
45 $\bar{1}$	0.454	10	45 $\bar{3}$	0.623	-
46 $\bar{1}$	0.512	70	413	0.357	185
47 $\bar{1}$	0.577	0	423	0.369	160
402	0.461	86	433	0.402	125
412	0.506	42	443	0.454	0
422	0.564	20	453	0.512	18
432	0.623	7	463	0.577	18
40 $\bar{2}$	0.350	34	473	0.647	-
41 $\bar{2}$	0.382	76	41 $\bar{3}$	0.480	38
42 $\bar{2}$	0.435	12	42 $\bar{3}$	0.461	2.5
43 $\bar{2}$	0.493	27	43 $\bar{3}$	0.461	65
44 $\bar{2}$	0.558	12.3	44 $\bar{3}$	0.473	20
45 $\bar{2}$	0.636	-	45 $\bar{3}$	0.499	50
412	0.337	6.5	46 $\bar{3}$	0.545	0
422	0.344	600	47 $\bar{3}$	0.597	7.5
432	0.376	260	404	0.571	0
442	0.422	400	414	0.616	5
452	0.480	80	40 $\bar{4}$	0.383	115
462	0.545	4.5	41 $\bar{4}$	0.402	305
472	0.592	0	42 $\bar{4}$	0.441	14.5
41 $\bar{2}$	0.428	26	43 $\bar{4}$	0.487	86
42 $\bar{2}$	0.408	170	44 $\bar{4}$	0.551	18
43 $\bar{2}$	0.415	3	45 $\bar{4}$	0.616	-
44 $\bar{2}$	0.434	22	414	0.383	58
45 $\bar{2}$	0.473	0	424	0.402	20
46 $\bar{2}$	0.525	10.8			
472	0.584	13.3			

TABLE III (Continued)

Year	1960	1961	Year	1960	1961
0.0	0.000	0.000	0.0	0.000	0.000
1.0	0.010	0.010	1.0	0.010	0.010
2.0	0.040	0.040	2.0	0.040	0.040
3.0	0.090	0.090	3.0	0.090	0.090
4.0	0.160	0.160	4.0	0.160	0.160
5.0	0.250	0.250	5.0	0.250	0.250
6.0	0.360	0.360	6.0	0.360	0.360
7.0	0.490	0.490	7.0	0.490	0.490
8.0	0.640	0.640	8.0	0.640	0.640
9.0	0.810	0.810	9.0	0.810	0.810
10.0	1.000	1.000	10.0	1.000	1.000
11.0	1.210	1.210	11.0	1.210	1.210
12.0	1.440	1.440	12.0	1.440	1.440
13.0	1.690	1.690	13.0	1.690	1.690
14.0	1.960	1.960	14.0	1.960	1.960
15.0	2.250	2.250	15.0	2.250	2.250
16.0	2.560	2.560	16.0	2.560	2.560
17.0	2.890	2.890	17.0	2.890	2.890
18.0	3.240	3.240	18.0	3.240	3.240
19.0	3.610	3.610	19.0	3.610	3.610
20.0	4.000	4.000	20.0	4.000	4.000
21.0	4.410	4.410	21.0	4.410	4.410
22.0	4.840	4.840	22.0	4.840	4.840
23.0	5.290	5.290	23.0	5.290	5.290
24.0	5.760	5.760	24.0	5.760	5.760
25.0	6.250	6.250	25.0	6.250	6.250
26.0	6.760	6.760	26.0	6.760	6.760
27.0	7.290	7.290	27.0	7.290	7.290
28.0	7.840	7.840	28.0	7.840	7.840
29.0	8.410	8.410	29.0	8.410	8.410
30.0	9.000	9.000	30.0	9.000	9.000
31.0	9.610	9.610	31.0	9.610	9.610
32.0	10.240	10.240	32.0	10.240	10.240
33.0	10.890	10.890	33.0	10.890	10.890
34.0	11.560	11.560	34.0	11.560	11.560
35.0	12.250	12.250	35.0	12.250	12.250
36.0	12.960	12.960	36.0	12.960	12.960
37.0	13.690	13.690	37.0	13.690	13.690
38.0	14.440	14.440	38.0	14.440	14.440
39.0	15.210	15.210	39.0	15.210	15.210
40.0	16.000	16.000	40.0	16.000	16.000
41.0	16.810	16.810	41.0	16.810	16.810
42.0	17.640	17.640	42.0	17.640	17.640
43.0	18.490	18.490	43.0	18.490	18.490
44.0	19.360	19.360	44.0	19.360	19.360
45.0	20.250	20.250	45.0	20.250	20.250
46.0	21.160	21.160	46.0	21.160	21.160
47.0	22.090	22.090	47.0	22.090	22.090
48.0	23.040	23.040	48.0	23.040	23.040
49.0	24.010	24.010	49.0	24.010	24.010
50.0	25.000	25.000	50.0	25.000	25.000

TABLE III (Continued)

Rel.			Rel.		
$hkl$	$\frac{\sin \theta}{\lambda}$	$ F_{hkl} ^2$	$hkl$	$\frac{\sin \theta}{\lambda}$	$ F_{hkl} ^2$
$\bar{4}34$	0.441	283	$\bar{4}16$	0.479	135
$\bar{4}44$	0.493	63	$\bar{4}26$	0.506	0
$\bar{4}54$	0.551	55	$\bar{4}36$	0.545	29
$\bar{4}64$	0.596	8.1	$\bar{4}46$	0.596	7.8
$\bar{4}1\bar{4}$	0.538	12.5	$\bar{4}2\bar{6}$	0.636	-
$\bar{4}2\bar{4}$	0.519	6.7	$\bar{4}3\bar{6}$	0.630	-
$\bar{4}3\bar{4}$	0.506	0	$\bar{4}4\bar{6}$	0.623	2.1
$\bar{4}4\bar{4}$	0.519	2.3	$\bar{4}5\bar{6}$	0.636	0
$\bar{4}5\bar{4}$	0.538	0			
$\bar{4}6\bar{4}$	0.577	0	$40\bar{7}$	0.525	21
$\bar{4}7\bar{4}$	0.623	0	$41\bar{7}$	0.525	0
			$42\bar{7}$	0.538	8.3
$405$	0.636	3	$43\bar{7}$	0.571	17
			$44\bar{7}$	0.609	6
$40\bar{5}$	0.422	4.5			
$41\bar{5}$	0.434	190	$\bar{4}17$	0.538	7.5
$42\bar{5}$	0.461	4	$\bar{4}27$	0.564	42
$43\bar{5}$	0.506	0	$\bar{4}37$	0.603	1.6
$44\bar{5}$	0.558	10			
$45\bar{5}$	0.623	-	$40\bar{8}$	0.584	9
			$41\bar{8}$	0.594	32
$\bar{4}15$	0.428	53	$42\bar{8}$	0.590	3.2
$\bar{4}25$	0.448	160	$43\bar{8}$	0.616	15.5
$\bar{4}35$	0.493	39	$44\bar{8}$	0.649	3.2
$\bar{4}45$	0.545	2.5			
$\bar{4}55$	0.598	-	$\bar{4}18$	0.591	0
			$\bar{4}28$	0.630	1.5
$\bar{4}1\bar{5}$	0.597	0			
$\bar{4}2\bar{5}$	0.577	10	$40\bar{9}$	0.592	0
$\bar{4}3\bar{5}$	0.564	11	$41\bar{9}$	0.649	2.3
$\bar{4}4\bar{5}$	0.571	35	$42\bar{9}$	0.636	-
$\bar{4}5\bar{5}$	0.584	0	$43\bar{9}$	0.649	-
$\bar{4}6\bar{5}$	0.616	0			
			$\bar{4}19$	0.649	0.6
$40\bar{6}$	0.467	130			
$41\bar{6}$	0.473	21	$500$	0.472	90
$42\bar{6}$	0.499	109	$510$	0.514	53.3
$43\bar{6}$	0.532	11.5	$520$	0.565	0
$44\bar{6}$	0.577	0	$530$	0.623	0
$45\bar{6}$	0.636	-			



[illegible]



TABLE III (Continued)

hk $\ell$	Rel.		hk $\ell$	Rel.	
	$\frac{\sin \theta}{\lambda}$	$ F_{hk\ell} ^2$		$\frac{\sin \theta}{\lambda}$	$ F_{hk\ell} ^2$
510	0.448	4.5	512	0.416	174
520	0.435	7.1	522	0.416	120
530	0.435	160	532	0.435	19
540	0.467	634	542	0.474	50
550	0.505	14.9	552	0.518	276
560	0.552	31.4	562	0.578	7
570	0.611	3.1	572	0.637	0
501	0.506	0	51 $\bar{2}$	0.520	0
511	0.552	0	52 $\bar{2}$	0.500	41
521	0.611	0	53 $\bar{2}$	0.493	240
50 $\bar{1}$	0.447	4	54 $\bar{2}$	0.506	0
51 $\bar{1}$	0.487	184	55 $\bar{2}$	0.537	4.1
52 $\bar{1}$	0.539	3	56 $\bar{2}$	0.572	0
53 $\bar{1}$	0.598	11.2	57 $\bar{2}$	0.624	-
511	0.422	90	503	0.601	2
521	0.416	16	513	0.643	0
531	0.429	195	50 $\bar{3}$	0.443	563
541	0.461	49	51 $\bar{3}$	0.461	8.1
551	0.505	26.6	52 $\bar{3}$	0.507	72.9
561	0.559	10.1	53 $\bar{3}$	0.559	0
571	0.617	0	54 $\bar{3}$	0.623	0
51 $\bar{1}$	0.474	28	513	0.422	278
52 $\bar{1}$	0.461	54	523	0.429	123
53 $\bar{1}$	0.461	16	533	0.455	21
54 $\bar{1}$	0.481	72	543	0.493	40
55 $\bar{1}$	0.516	0	553	0.542	19
56 $\bar{1}$	0.559	16.7	563	0.598	6.8
57 $\bar{1}$	0.611	38.1	51 $\bar{3}$	0.565	7.7
502	0.550	0	52 $\bar{3}$	0.546	0
512	0.598	0	53 $\bar{3}$	0.533	25
50 $\bar{2}$	0.433	18	54 $\bar{3}$	0.539	0
512	0.467	31	55 $\bar{3}$	0.571	18.8
522	0.520	33	56 $\bar{3}$	0.598	-
532	0.572	16	57 $\bar{3}$	0.640	-
542	0.637	-	534	0.559	0

(continued) THE GREAT

Year	1940	1941	1942	1943	1944	1945	1946	1947	1948	1949	1950	1951	1952	1953	1954	1955	1956	1957	1958	1959	1960	1961	1962	1963	1964	1965	1966	1967	1968	1969	1970	1971	1972	1973	1974	1975	1976	1977	1978	1979	1980	1981	1982	1983	1984	1985	1986	1987	1988	1989	1990	1991	1992	1993	1994	1995	1996	1997	1998	1999	2000	2001	2002	2003	2004	2005	2006	2007	2008	2009	2010	2011	2012	2013	2014	2015	2016	2017	2018	2019	2020	2021	2022	2023	2024	2025	2026	2027	2028	2029	2030	2031	2032	2033	2034	2035	2036	2037	2038	2039	2040	2041	2042	2043	2044	2045	2046	2047	2048	2049	2050	2051	2052	2053	2054	2055	2056	2057	2058	2059	2060	2061	2062	2063	2064	2065	2066	2067	2068	2069	2070	2071	2072	2073	2074	2075	2076	2077	2078	2079	2080	2081	2082	2083	2084	2085	2086	2087	2088	2089	2090	2091	2092	2093	2094	2095	2096	2097	2098	2099	2100
1940	1941	1942	1943	1944	1945	1946	1947	1948	1949	1950	1951	1952	1953	1954	1955	1956	1957	1958	1959	1960	1961	1962	1963	1964	1965	1966	1967	1968	1969	1970	1971	1972	1973	1974	1975	1976	1977	1978	1979	1980	1981	1982	1983	1984	1985	1986	1987	1988	1989	1990	1991	1992	1993	1994	1995	1996	1997	1998	1999	2000	2001	2002	2003	2004	2005	2006	2007	2008	2009	2010	2011	2012	2013	2014	2015	2016	2017	2018	2019	2020	2021	2022	2023	2024	2025	2026	2027	2028	2029	2030	2031	2032	2033	2034	2035	2036	2037	2038	2039	2040	2041	2042	2043	2044	2045	2046	2047	2048	2049	2050	2051	2052	2053	2054	2055	2056	2057	2058	2059	2060	2061	2062	2063	2064	2065	2066	2067	2068	2069	2070	2071	2072	2073	2074	2075	2076	2077	2078	2079	2080	2081	2082	2083	2084	2085	2086	2087	2088	2089	2090	2091	2092	2093	2094	2095	2096	2097	2098	2099	2100	

TABLE III (Continued)

Rel.			Rel.		
$hkl$	$\frac{\sin \theta}{\lambda}$	$ F_{hkl} ^2$	$hkl$	$\frac{\sin \theta}{\lambda}$	$ F_{hkl} ^2$
504	0.447	380	516	0.513	130
514	0.474	103	526	0.533	21
524	0.510	165	536	0.565	8
534	0.559	0	546	0.611	0
544	0.617	0			
			507	0.551	0
514	0.442	0	517	0.558	0
524	0.455	2.5	527	0.584	-
534	0.481	60	537	0.611	-
544	0.520	3			
554	0.575	6	517	0.558	0
564	0.637	0	527	0.585	7.5
			537	0.617	2.2
514	0.618	3			
524	0.580	44	508	0.601	14
534	0.624	0.8	518	0.603	-
544	0.580	0	528	0.624	0
554	0.609	0	538	0.646	-
564	0.630	-			
			518	0.617	0
505	0.473	21	528	0.643	0
515	0.494	16.5			
525	0.525	0	600	0.565	6.3
535	0.565	-	610	0.604	3.6
545	0.617	8.7			
			610	0.539	0
515	0.474	95	620	0.519	3.2
525	0.490	148	630	0.520	0
535	0.520	18	640	0.533	0
545	0.565	16	650	0.572	4.5
555	0.613	3.5	660	0.607	7.6
535	0.636	4	601	0.600	6.3
545	0.636	3.6	611	0.643	0
506	0.508	1	601	0.540	9.3
516	0.520	200	611	0.578	10.9
526	0.545	0	621	0.630	-
536	0.585	-			
546	0.630	-	611	0.513	60
			621	0.506	152







TABLE III (Continued)

hkl	$\frac{\sin \theta}{\lambda}$	Rel.	hkl	$\frac{\sin \theta}{\lambda}$	Rel.
		$ F_{hkl} ^2$			$ F_{hkl} ^2$
$\bar{5}31$	0.506	0	$\bar{5}53$	0.591	7
$\bar{6}41$	0.526	0	$\bar{6}33$	0.615	5
$\bar{6}51$	0.565	12	$\bar{6}53$	0.600	3
$\bar{6}61$	0.603	0	$\bar{6}63$	0.633	17
$\bar{6}1\bar{1}$	0.565	40	$60\bar{4}$	0.524	2.8
$\bar{6}2\bar{1}$	0.552	0	$61\bar{2}$	0.552	0
$\bar{6}3\bar{1}$	0.552	1.7	$62\bar{4}$	0.592	-
$\bar{6}4\bar{1}$	0.552	2.5	$63\bar{4}$	0.636	12
$\bar{6}5\bar{1}$	0.585	8	$\bar{6}14$	0.513	6.5
$\bar{6}6\bar{1}$	0.615	5	$\bar{6}24$	0.519	102
$602$	0.640	0	$\bar{6}34$	0.532	3.5
$60\bar{2}$	0.524	0	$\bar{6}44$	0.565	0
$61\bar{2}$	0.558	0	$\bar{6}54$	0.611	0
$62\bar{2}$	0.611	0	$60\bar{5}$	0.540	0
$\bar{6}12$	0.500	90	$61\bar{5}$	0.565	0
$\bar{6}22$	0.500	0	$62\bar{5}$	0.598	-
$\bar{6}32$	0.506	0	$63\bar{5}$	0.636	-
$\bar{6}42$	0.529	0	$\bar{6}15$	0.532	0
$\bar{6}52$	0.572	8.9	$\bar{6}25$	0.546	8.6
$\bar{6}62$	0.613	0	$\bar{6}35$	0.565	2.5
$\bar{6}1\bar{2}$	0.604	2.4	$\bar{6}45$	0.597	15
$\bar{6}2\bar{2}$	0.592	-	$60\bar{6}$	0.566	1
$\bar{6}3\bar{2}$	0.578	-	$61\bar{6}$	0.585	0
$\bar{6}4\bar{2}$	0.581	2.6	$62\bar{6}$	0.617	-
$\bar{6}5\bar{2}$	0.591	0	$63\bar{6}$	0.649	-
$\bar{6}6\bar{2}$	0.635	0	$\bar{6}16$	0.565	5
$60\bar{3}$	0.518	4.9	$\bar{6}26$	0.578	0
$61\bar{3}$	0.552	0	$\bar{6}36$	0.604	9
$62\bar{3}$	0.593	0	$\bar{6}46$	0.640	0
$63\bar{3}$	0.643	4	$60\bar{7}$	0.601	2.6
$\bar{6}13$	0.500	50	$61\bar{7}$	0.617	-
$\bar{6}23$	0.500	20	$62\bar{7}$	0.647	-
$\bar{6}33$	0.513	138			
$\bar{6}43$	0.546	10			

TABLE III (Continued)

Left			Right		
$\lambda$	$\frac{P_{010}}{\lambda}$	$\lambda$	$\lambda$	$\frac{P_{010}}{\lambda}$	$\lambda$
1	0.000	100	0	0.000	100
2	0.000	100	0	0.000	100
3	0.000	100	0	0.000	100
4	0.000	100	0	0.000	100
5	0.000	100	0	0.000	100
6	0.000	100	0	0.000	100
7	0.000	100	0	0.000	100
8	0.000	100	0	0.000	100
9	0.000	100	0	0.000	100
10	0.000	100	0	0.000	100
11	0.000	100	0	0.000	100
12	0.000	100	0	0.000	100
13	0.000	100	0	0.000	100
14	0.000	100	0	0.000	100
15	0.000	100	0	0.000	100
16	0.000	100	0	0.000	100
17	0.000	100	0	0.000	100
18	0.000	100	0	0.000	100
19	0.000	100	0	0.000	100
20	0.000	100	0	0.000	100
21	0.000	100	0	0.000	100
22	0.000	100	0	0.000	100
23	0.000	100	0	0.000	100
24	0.000	100	0	0.000	100
25	0.000	100	0	0.000	100
26	0.000	100	0	0.000	100
27	0.000	100	0	0.000	100
28	0.000	100	0	0.000	100
29	0.000	100	0	0.000	100
30	0.000	100	0	0.000	100
31	0.000	100	0	0.000	100
32	0.000	100	0	0.000	100
33	0.000	100	0	0.000	100
34	0.000	100	0	0.000	100
35	0.000	100	0	0.000	100
36	0.000	100	0	0.000	100
37	0.000	100	0	0.000	100
38	0.000	100	0	0.000	100
39	0.000	100	0	0.000	100
40	0.000	100	0	0.000	100
41	0.000	100	0	0.000	100
42	0.000	100	0	0.000	100
43	0.000	100	0	0.000	100
44	0.000	100	0	0.000	100
45	0.000	100	0	0.000	100
46	0.000	100	0	0.000	100
47	0.000	100	0	0.000	100
48	0.000	100	0	0.000	100
49	0.000	100	0	0.000	100
50	0.000	100	0	0.000	100
51	0.000	100	0	0.000	100
52	0.000	100	0	0.000	100
53	0.000	100	0	0.000	100
54	0.000	100	0	0.000	100
55	0.000	100	0	0.000	100
56	0.000	100	0	0.000	100
57	0.000	100	0	0.000	100
58	0.000	100	0	0.000	100
59	0.000	100	0	0.000	100
60	0.000	100	0	0.000	100
61	0.000	100	0	0.000	100
62	0.000	100	0	0.000	100
63	0.000	100	0	0.000	100
64	0.000	100	0	0.000	100
65	0.000	100	0	0.000	100
66	0.000	100	0	0.000	100
67	0.000	100	0	0.000	100
68	0.000	100	0	0.000	100
69	0.000	100	0	0.000	100
70	0.000	100	0	0.000	100
71	0.000	100	0	0.000	100
72	0.000	100	0	0.000	100
73	0.000	100	0	0.000	100
74	0.000	100	0	0.000	100
75	0.000	100	0	0.000	100
76	0.000	100	0	0.000	100
77	0.000	100	0	0.000	100
78	0.000	100	0	0.000	100
79	0.000	100	0	0.000	100
80	0.000	100	0	0.000	100
81	0.000	100	0	0.000	100
82	0.000	100	0	0.000	100
83	0.000	100	0	0.000	100
84	0.000	100	0	0.000	100
85	0.000	100	0	0.000	100
86	0.000	100	0	0.000	100
87	0.000	100	0	0.000	100
88	0.000	100	0	0.000	100
89	0.000	100	0	0.000	100
90	0.000	100	0	0.000	100
91	0.000	100	0	0.000	100
92	0.000	100	0	0.000	100
93	0.000	100	0	0.000	100
94	0.000	100	0	0.000	100
95	0.000	100	0	0.000	100
96	0.000	100	0	0.000	100
97	0.000	100	0	0.000	100
98	0.000	100	0	0.000	100
99	0.000	100	0	0.000	100
100	0.000	100	0	0.000	100

TABLE III (Continued)

hkl	Rel.		hkl	Rel.	
	$\frac{\sin \theta}{\lambda}$	$ F_{hkl} ^2$		$\frac{\sin \theta}{\lambda}$	$ F_{hkl} ^2$
$\bar{6}17$	0.604	0	$70\bar{3}$	0.606	6.5
$\bar{6}27$	0.624	6.3	$71\bar{3}$	0.637	52.6
$\bar{6}37$	0.646	-	$74\bar{3}$	0.618	17
$60\bar{8}$	0.641	0	$\bar{7}13$	0.618	17
$\bar{6}18$	0.649	-	$\bar{7}23$	0.585	0
$750$	0.636	24.8	$\bar{7}33$	0.585	14
$\bar{7}30$	0.610	15	$\bar{7}43$	0.585	3.2
$70\bar{1}$	0.634	0	$\bar{7}53$	0.604	12
$\bar{7}11$	0.604	0	$70\bar{4}$	0.606	19
$\bar{7}21$	0.598	5	$71\bar{4}$	0.633	-
$\bar{7}31$	0.591	7	$\bar{7}14$	0.591	18.9
$\bar{7}41$	0.604	0	$\bar{7}24$	0.591	4
$\bar{7}51$	0.622	14	$\bar{7}34$	0.604	10
$\bar{7}3\bar{1}$	0.630	0	$70\bar{5}$	0.615	48.7
$70\bar{2}$	0.615	13.3	$75\bar{1}$	0.640	-
$\bar{7}12$	0.591	0	$\bar{7}15$	0.604	10.6
$\bar{7}22$	0.585	0	$\bar{7}25$	0.610	0
$\bar{7}32$	0.585	0	$\bar{7}35$	0.623	0
$\bar{7}42$	0.597	0	$\bar{7}45$	0.649	0
			$70\bar{6}$	0.633	2.9
			$\bar{7}16$	0.627	38.4
			$\bar{7}26$	0.637	25.0

The factors by which relative  $F^2$  values of a given film were multiplied to reach the common level are:



TABLE III (Continued)

Year	Relative $\frac{W}{A}$	Year	Relative $\frac{W}{A}$	Year
1957	0.000	1967	0.000	1977
1958	0.000	1968	0.000	1978
1959	0.000	1969	0.000	1979
1960	0.000	1970	0.000	1980
1961	0.000	1971	0.000	1981
1962	0.000	1972	0.000	1982
1963	0.000	1973	0.000	1983
1964	0.000	1974	0.000	1984
1965	0.000	1975	0.000	1985
1966	0.000	1976	0.000	1986
1967	0.000	1977	0.000	1987
1968	0.000	1978	0.000	1988
1969	0.000	1979	0.000	1989
1970	0.000	1980	0.000	1990
1971	0.000	1981	0.000	1991
1972	0.000	1982	0.000	1992
1973	0.000	1983	0.000	1993
1974	0.000	1984	0.000	1994
1975	0.000	1985	0.000	1995
1976	0.000	1986	0.000	1996
1977	0.000	1987	0.000	1997
1978	0.000	1988	0.000	1998
1979	0.000	1989	0.000	1999
1980	0.000	1990	0.000	2000
1981	0.000	1991	0.000	2001
1982	0.000	1992	0.000	2002
1983	0.000	1993	0.000	2003
1984	0.000	1994	0.000	2004
1985	0.000	1995	0.000	2005
1986	0.000	1996	0.000	2006
1987	0.000	1997	0.000	2007
1988	0.000	1998	0.000	2008
1989	0.000	1999	0.000	2009
1990	0.000	2000	0.000	2010
1991	0.000	2001	0.000	2011
1992	0.000	2002	0.000	2012
1993	0.000	2003	0.000	2013
1994	0.000	2004	0.000	2014
1995	0.000	2005	0.000	2015
1996	0.000	2006	0.000	2016
1997	0.000	2007	0.000	2017
1998	0.000	2008	0.000	2018
1999	0.000	2009	0.000	2019
2000	0.000	2010	0.000	2020

The factors of which relative  $\frac{W}{A}$  values of a given

Year were multiplied to reach the common level are:



<u>Axis</u>	<u>Layer</u>	<u>Factor</u>	<u>Axis</u>	<u>Layer</u>	<u>Factor</u>
$a_1$	1	1.20	$a_3''$	0	2.05
	2	0.99		1	2.35
	3	1.05		2	2.56
	4	1.46		3	2.78
$a_2$	0	0.56		4	2.55
	1	0.91		5	3.04
	2	1.29			
	3	1.32			
	4	1.62			

Absorption effects were calculated to be negligible.

Factor	Level	Mean	Factor	Level	Mean
1	1	1.70	1	1	1.70
2	2	0.99	2	2	0.99
3	3	1.05	3	3	1.05
4	4	1.46	4	4	1.46
5	5	0.50	5	5	0.50
6	6	1.71	6	6	1.71
7	7	1.22	7	7	1.22
8	8	1.01	8	8	1.01

Assumption effects were calculated to be negligible.

The effects of the factors were calculated to be negligible.

## DETERMINATION OF PARAMETERS

The atomic parameters listed by Broge for the compound cell were converted by graphical means to the coordinates in the primitive unit as follows:

	x	y	z
C <sub>1</sub>	-0.139	+0.044	-0.057
C <sub>2</sub>	-0.198	-0.024	-0.259
C <sub>3</sub>	-0.224	-0.266	-0.204
C <sub>4</sub>	-0.133	-0.170	0.000
F <sub>1</sub>	+0.220	-0.229	0.000
F <sub>2</sub>	-0.116	+0.115	-0.454
F <sub>3</sub>	-0.487	+0.030	-0.364
F <sub>4</sub>	-0.083	-0.415	-0.354
F <sub>5</sub>	-0.374	-0.479	-0.268
F <sub>6</sub>	-0.244	-0.354	+0.180

Figures 13 and 14 show projections on (010) and (100) of the reduced cell that would be obtained using the above. The centrosymmetric model is clearly outlined.

Amplitudes calculated from these positions however failed to agree with observed values. This indicated that the proposed structure was not close to the true one and that the experimental data should be used systematically in a search for the correct structure.

The atomic parameters listed by Rogers for the same  
 point were converted by graphical means to the  
 coordinates in the primitive cell as follows:

x	y	z	
-0.007	+0.004	-0.100	C1
-0.008	-0.004	-0.100	C2
-0.004	-0.004	-0.024	C3
0.000	-0.007	-0.100	C4
0.000	-0.007	+0.020	V1
-0.004	+0.004	-0.100	V2
-0.004	+0.000	-0.007	V3
-0.004	-0.004	-0.000	V4
-0.008	-0.007	-0.024	V5
-0.100	-0.004	-0.004	V6

Figures 13 and 14 show projections on (010) and (100)  
 of the model which would be obtained using the above.  
 The centrosymmetric model is clearly outlined.  
 Significant evidence from these positions however  
 failed to agree with observed values. This indicated  
 that the proposed structure was not close to the true  
 one and that the experimental data should be used system-  
 atically in a search for the correct structure.



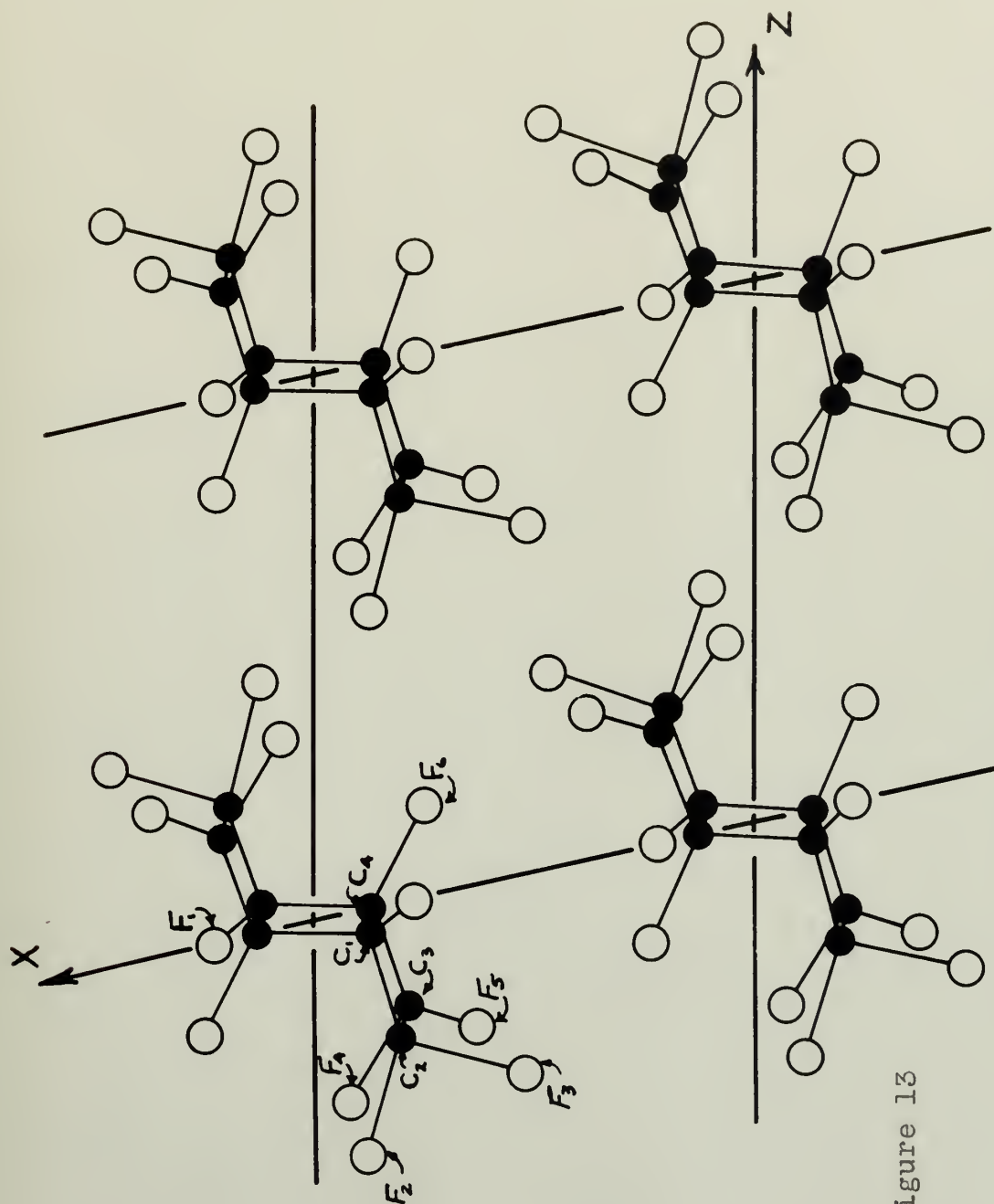
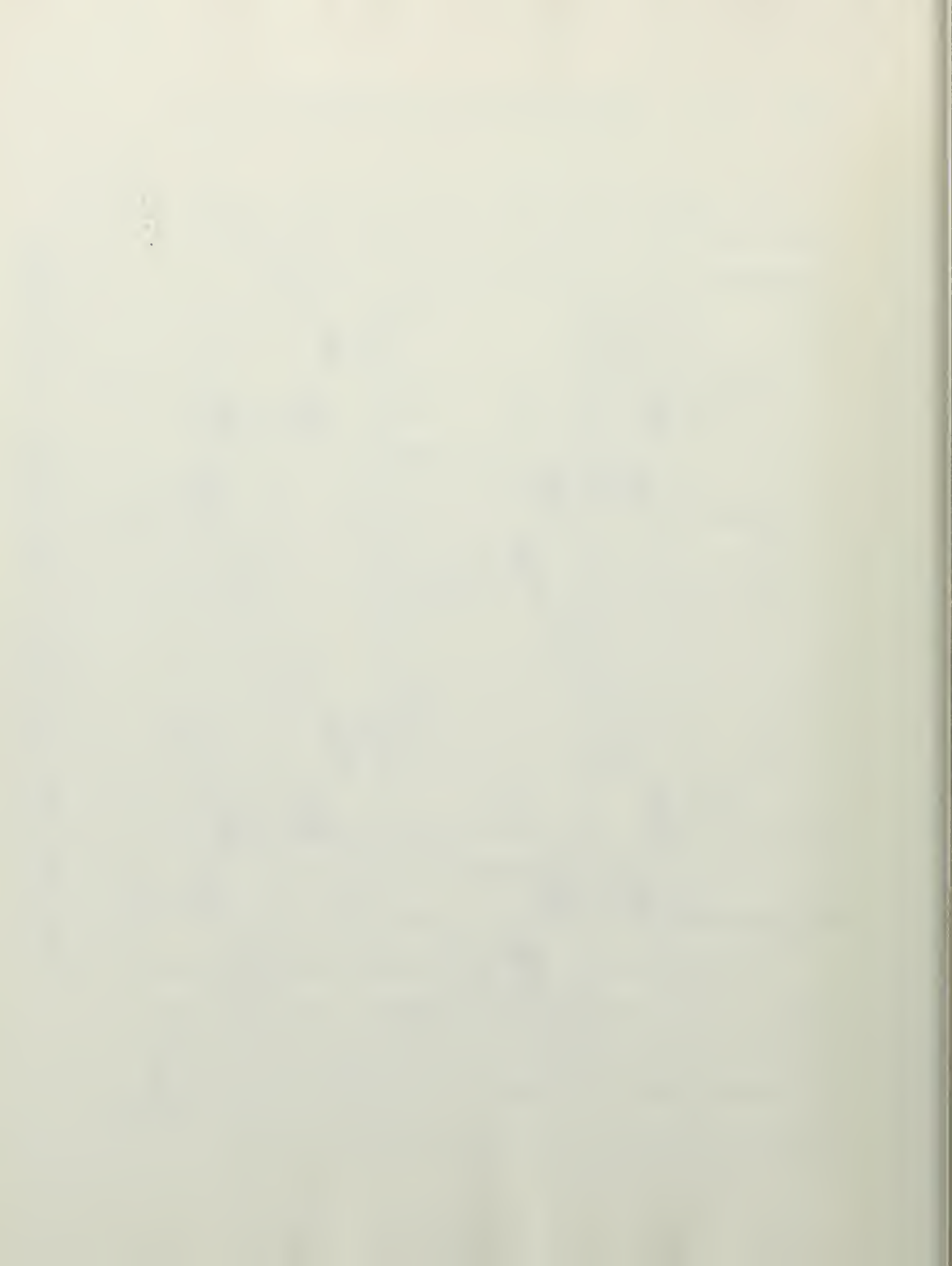


Figure 13

PROJECTION ON (010) FROM DATA OF BROGE



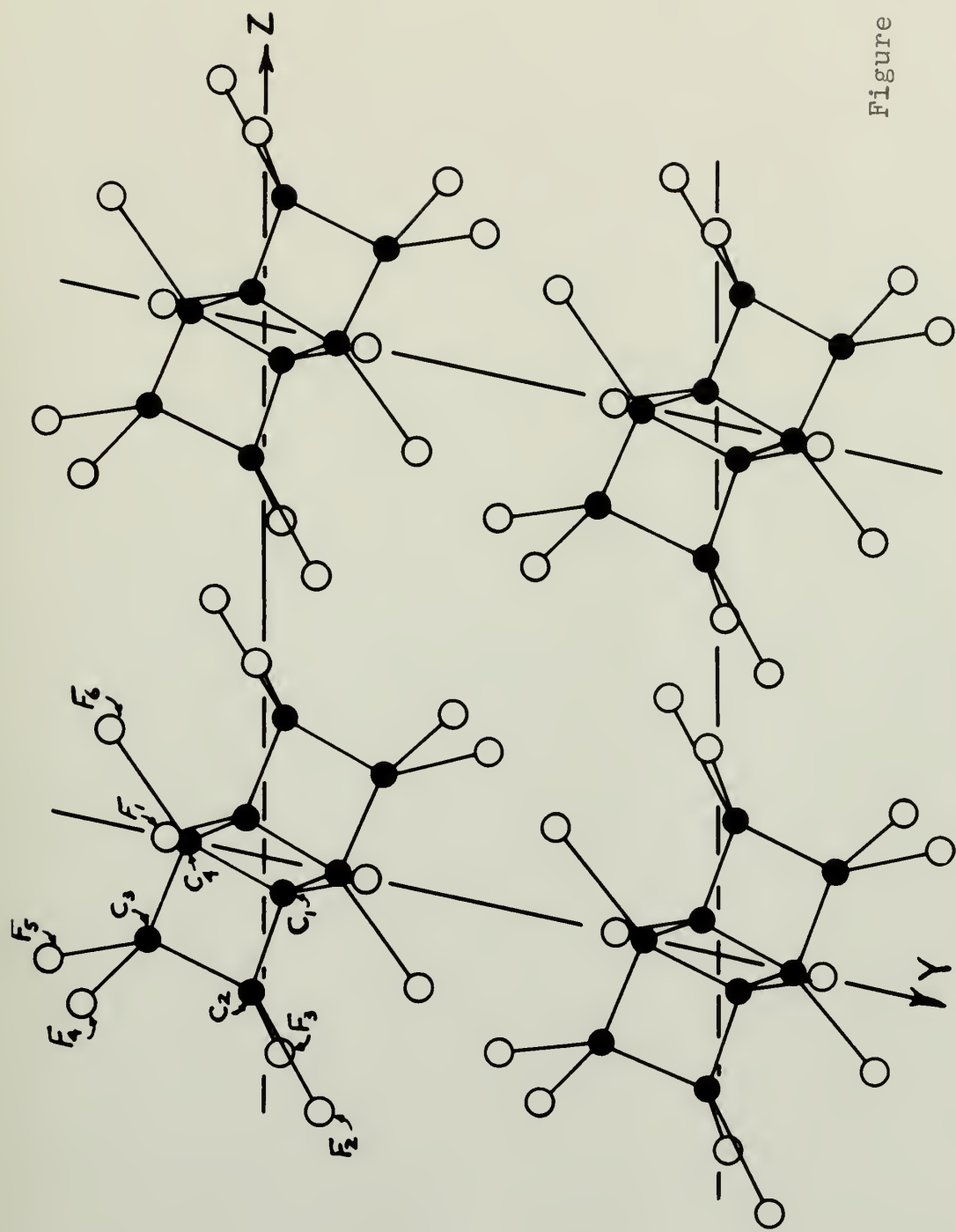


Figure 14

PROJECTION ON (100) FROM DATA OF BROGE





Patterson Projection,  $P(Y,Z)$ .

Since the first data were observed for rotation about  $a_1$  the Patterson projection on (100) was computed. It can be shown that

$$\begin{aligned}
 KP(Y,Z) = & \sum_{k=0}^{\infty} \sum_{\ell=0}^{\infty} \left[ |F_{0k\ell}|^2 + |F_{0\bar{k}\ell}|^2 \right] \cos 2 \pi k Y \cos 2 \pi \ell Z \\
 & - \sum_{k=1}^{\infty} \sum_{\ell=1}^{\infty} \left[ |F_{0k\ell}|^2 - |F_{0\bar{k}\ell}|^2 \right] \sin 2 \pi k Y \sin 2 \pi \ell Z
 \end{aligned}$$

(neglecting constant additive terms).

The projection is shown in Figure 15. Contours start at 0 and are drawn at intervals of 50 in relative Patterson density. Aside from the identity peak at the origin, a single solid ridge is seen extending from  $Y=0, Z=0$  to  $Y=1, Z=-1/2$  with peaks at intervals along the entire length. This would lead one to believe that the long axis of the molecule lay parallel to the ridge. Further evidence lies in the fact that the (012) reflection is indicative of strong temperature diffuse scattering. The significance of such reflections is discussed by Lonsdale.<sup>(22)</sup> Trial parameters assigned to the atoms on the basis of such an orientation failed to give a satisfactory comparison of calculated and observed amplitudes for  $F(00\ell)$  and  $F(0k0)$ .

pattern projection  $P(Y, Z)$ .

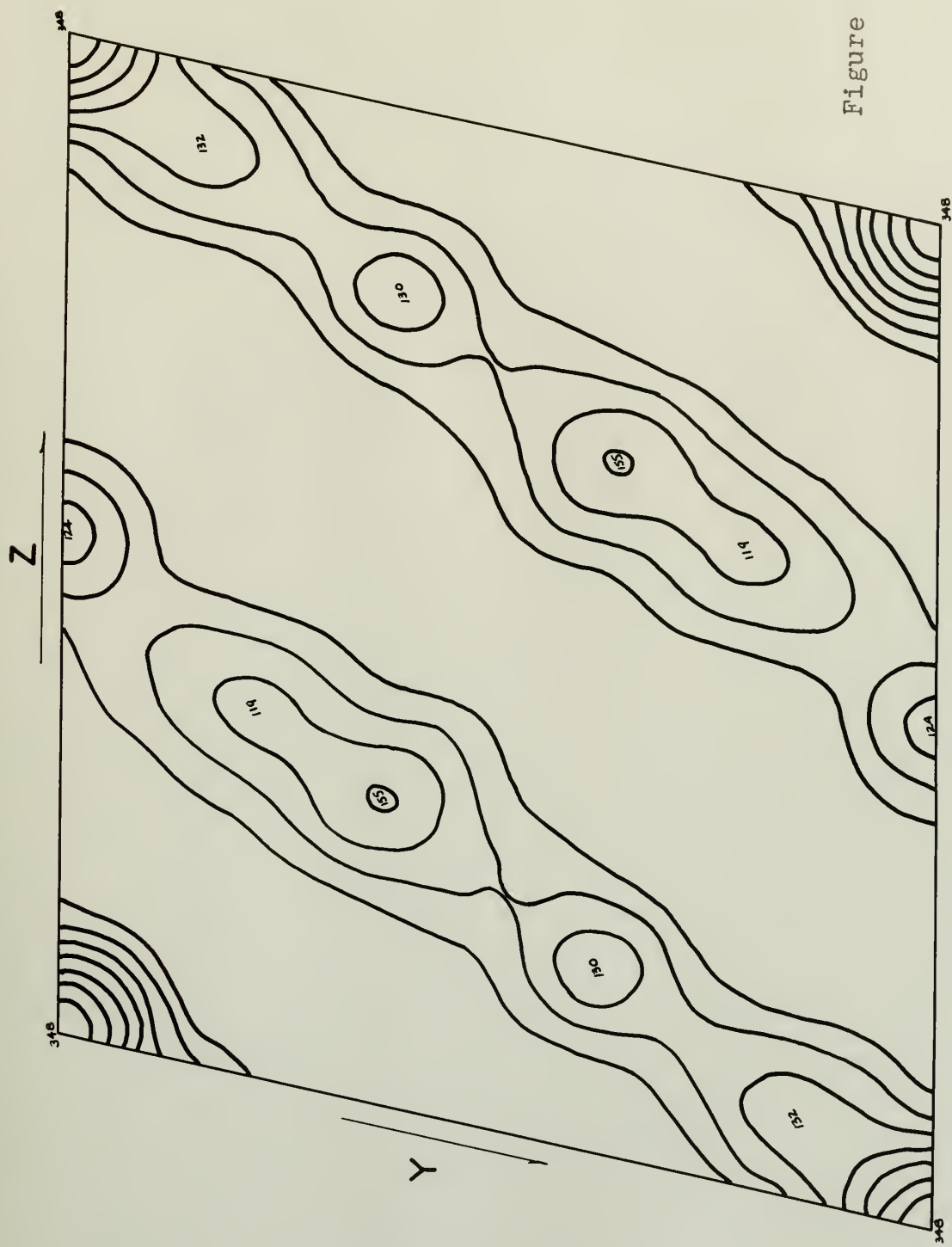
Since the first data were observed for rotation about  $z$ , the pattern projection on (100) was computed. It can be shown that

$$P(Y, Z) = \sum_{k=0}^{\infty} \sum_{l=0}^{\infty} \left[ \frac{1}{2} (|x_{0k}|^2 + |x_{0l}|^2) \cos 2\pi k Y \cos 2\pi l Z \right]$$

$$- \sum_{k=1}^{\infty} \sum_{l=1}^{\infty} \left[ \frac{1}{2} (|x_{0k}|^2 - |x_{0l}|^2) \sin 2\pi k Y \sin 2\pi l Z \right]$$

(neglecting constant additive terms).

The projection is shown in Figure 10. Contours start at 0 and are drawn at intervals of 50 in relative Patterson density. Aside from the identity peak at the origin, a single ridge is seen extending from  $Y=0, Z=0$  to  $Y=1, Z=1/2$  with peaks at intervals along the entire length. This would lead one to believe that the long axis of the molecule lay parallel to the ridge. Further evidence lies in the fact that the (011) reflection is indicative of strong temperature diffuse scattering. The slight-ness of such reflections is discussed by Lonsdale.<sup>(22)</sup> Total parameters assigned to the atoms on the basis of such an orientation failed to give a satisfactory comparison of calculated and observed amplitudes for  $P(001)$  and  $P(010)$ .



PATTERSON PROJECTION  $P(Y,Z)$

Figure 15





Patterson Projection,  $P(X,Z)$ .

The function, apart from constant additive terms, may be expressed as:

$$\begin{aligned}
 KP(X,Z) = & \sum_{h=0}^{\infty} \sum_{\ell=0}^{\infty} \left[ |F_{h0\ell}|^2 + |F_{\bar{h}0\ell}|^2 \right] \cos 2 \pi hX \cos 2 \pi \ell Z \\
 & - \sum_{h=1}^{\infty} \sum_{\ell=1}^{\infty} \left[ |F_{h0\ell}|^2 - |F_{\bar{h}0\ell}|^2 \right] \sin 2 \pi hX \sin 2 \pi \ell Z.
 \end{aligned}$$

The projection on (010) of the primitive reduced cell is shown in Figure 16. Contours are drawn as before. Several isolated peaks appear and may be grouped along straight lines with striking regularity. Several orientations based on features of this projection were tried but failed to give satisfactory agreement of observed and calculated amplitudes.

Peaks arising from vectors between equivalent atoms will appear on  $P(X,Z)$  at  $X=2x, Z=2z$ . A scaled model of the unit cell was made so that trial structures could be set up with cork balls to represent appropriate atoms. Even with the model it was not possible to determine which of the peaks appeared because of equivalent atoms. The parameters obtained from Broge's analysis were used to plot peaks at  $2x, 2z$  on an overlay of the Patterson projection. The coordinates were adjusted to agree with the peaks on the projection and structure amplitudes

Projection,  $V(x, z)$ .

The function, apart from constant additive terms,

may be expressed as:

$$V(x, z) = \sum_{n=0}^{\infty} \sum_{m=0}^{\infty} \frac{1}{n!m!} \left[ \frac{\partial^2}{\partial x^2 \partial z^2} \right] V(x, z) \cos 2\pi n x \cos 2\pi m z$$

$$= \sum_{n=1}^{\infty} \sum_{m=1}^{\infty} \frac{1}{n!m!} \left[ \frac{\partial^2}{\partial x^2 \partial z^2} \right] V(x, z) \sin 2\pi n x \sin 2\pi m z$$

The projection on (010) of the primitive reduced cell is shown in figure 1. Contours are drawn as before. Several isolated peaks appear and may be grouped along straight lines with striking regularity. Several extensions based on features of this projection were tried but failed to give satisfactory agreement of observed and calculated amplitudes.

Peaks arising from vectors between equivalent atoms will appear on  $V(x, z)$  at  $x=0, z=0$ . A scaled model of the unit cell was made so that trial structures could be set up with cork balls to represent appropriate atoms. Even with the model it was not possible to determine which of the peaks appeared because of equivalent atoms. The parameters obtained from Brogo's analysis were used to plot peaks at  $2x, 2z$  on an overlay of the projection projection. The coordinates were adjusted to agree with the peaks on the projection and structure amplitudes







computed. Again there was no agreement.

Since the ( $\bar{2}21$ ) reflection was so very strong it was suspected that a large amount of scattering material was located in the corresponding plane. The Patterson density from  $P(X,Z)$  was plotted on a properly drawn grid of the ( $\bar{2}21$ ) plane. Any vectors between atoms lying in this plane would appear in their true length in the preceding plot. The scaled molecular model was placed in several orientations such that many of the fluorines lay in the plane. No vectors could be recognized however.

Patterson Projection,  $P(X',Z')$ .

The projection on the  $a_1'' a_3''$  face of the unit cell of crystal #4 was calculated. This corresponds to  $(01\bar{1})$  of the reduced unit. It is shown in Figure 17. Contours start at 0 and are drawn at intervals of 20 in arbitrary relative Patterson density. Here again there is a long ridge extending from  $X'=0, Z'=0$  to  $X'=1/2, Z'=1$  with poorly defined peaks all along the ridge. The plane of projection was properly fitted into the primitive reduced cell to see if three coordinates could be assigned to any one vector peak. This procedure failed to clarify the situation however.

computed. Again there was no agreement.

Since the (111) reflection was so very strong it was suspected that a large amount of scattering material was located in the corresponding plane. The Patterson density from  $P(x, y)$  was plotted as a properly drawn grid of the (111) plane. Any vectors between atoms lying in this plane would appear in their true length in the projecting plot. The scaled molecular model was placed in several orientations such that many of the fluorine lay in the plane. No vectors could be recognized however.

Patterson projection,  $P(x, y)$ .

The projection on the  $xy$  face of the unit cell of crystal was calculated. This corresponds to (011) of the reduced unit. It is shown in Figure 17. Contours start at 0 and are drawn at intervals of 20 in arbitrary relative Patterson density. There again there is a long ridge extending from  $x=0, y=0$  to  $x=1/2, y=1/2$  with peaks defined along the ridge. The plane of projection was properly tilted into the perspective reduced cell to see if three coordinates could be assigned to any one vector peak. This procedure failed to clarify the situation however.

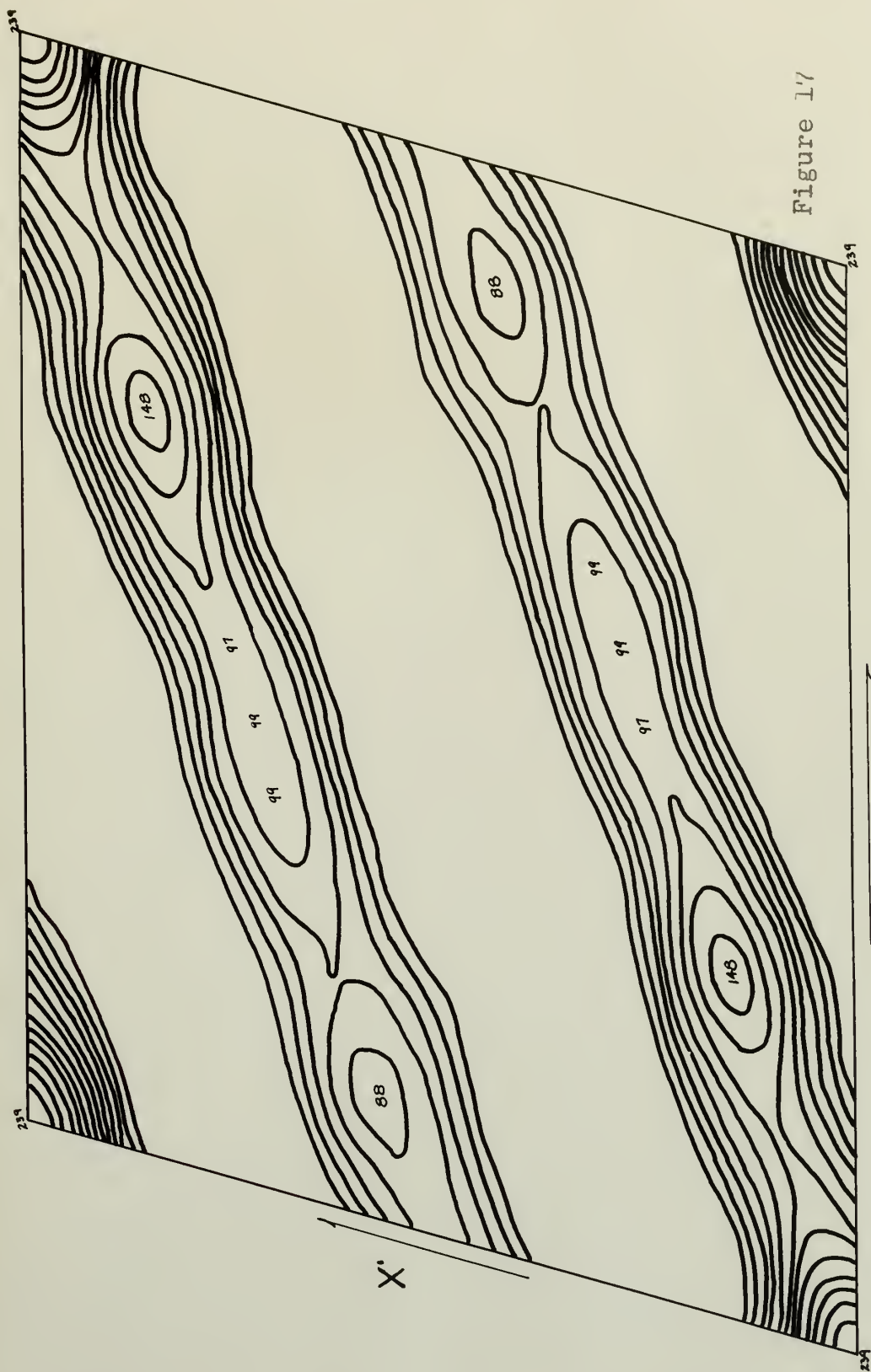


Figure 17

PATTERSON PROJECTION  $P(X', Z')$





### Phase Inequalities and Unitary Structure Amplitudes.

Since none of the projections were successfully interpreted, the possibility of phase determination by the recently proposed inequality procedure was explored. Unitary amplitudes are required in the inequality method and were, therefore, calculated for  $(0k\ell)$  and  $(h0\ell)$  data.

It was soon clear that only a few F's were greater than 0.5 and that there were certainly not enough of sufficiently high magnitude to determine more than a few phases. If the assumed model were correct, the method might be further exploited. However, if the center of symmetry is lost, the procedure no longer is applicable.

### Temporary Conclusion of the Work

Thus far, all attempts at parameter determination had failed. It was at this point that  $C_4Cl_8$  became available for study. It was decided that more would be accomplished in the long run if the  $C_8F_{12}$  problem were dropped at this time in order to determine the structure of the octachlorocyclobutane. Any knowledge gained concerning the configuration of the carbon ring in the latter compound might then lead to reconsideration of the proper model to use in work on the former.

Recently Professor Miller made available for study the compound  $C_8Cl_4F_8$  analagous to  $C_8F_{12}$  wherein the

board inequalities and binary structure inequalities.  
 Since some of the projections were successfully  
 interpreted, the possibility of phase determination by  
 the recently proposed inequality procedure was explored.  
 Binary inequalities are reported in the inequality method  
 and were, therefore, calculated for (001) and (100) data.  
 It was also clear that only a few  $F^2$ 's were greater

than 0.5 and that there were certainly not enough of  
 sufficiently high magnitude to determine more than a few  
 phases. If the assumed model were correct, the method  
 might be further exploited. However, if the center of  
 symmetry is lost, the procedure no longer is applicable.

#### Temporary Conclusion of the Work

Thus far, all attempts at parameter determination  
 had failed. It was at this point that  $C_{4v}$  became  
 available for study. It was decided that more would be  
 accomplished in the long run if the  $C_{4v}$  problem were  
 dropped at this time in order to determine the structure  
 of the octahedral complex. Any knowledge gained con-  
 cerning the configuration of the cation ring in the latter  
 compound might then lead to reconsideration of the proper  
 model to use in work on the former.

Recently Professor Miller made available for study

the compound  $C_{4v}$  analogous to  $C_{4v}$  wherein the

fluorines attached to the central carbon ring have been replaced by chlorine atoms. It was at first believed that structure studies of this material might help in the solution of the hexafluorobutadiene dimer. Preliminary work has shown that crystals of the new compound have cubic symmetry and thus are not isomorphous with  $C_8F_{12}$ . There is, however, evidence of molecular rotation at room temperatures so further work at lower temperatures may give more information.



Fluorine attached to the central carbon they have been replaced by chlorine atoms. It was at first believed that structural studies of this material might help in the solution of the hexafluorobenzene puzzle. Further study has shown that crystals of the new compound have cubic symmetry and thus are not isomorphous with C<sub>6</sub>F<sub>6</sub>. There is, however, evidence of retained rotation at room temperature so further work at lower temperatures may give more information.



## SUGGESTIONS FOR FURTHER STUDY

In view of the evidence of the non-planar carbon ring in  $C_4Cl_8$  it may be possible that the rings in  $C_8F_{12}$  are puckered with a resulting loss of a center of symmetry for the molecule. If this be true, the structure problem will be extremely difficult but not impossible.

Nearly complete  $(hk\ell)$  data have been obtained for the primitive reduced cell. It is thus possible to calculate a series of three-dimensional Patterson sections for small increments along a chosen axis, probably  $a_2$ . From these sections enough vectors might be resolved to lead to a satisfactory structure determination.

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King in 1961. It was in 1962 that the

$2\text{Mg}^{2+}$  are released with a resulting loss of a cation.

to symmetry for the molecule. If this is true, the

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impossible. In 1990, the first of these was published.

Manuscript accepted 18 July 2005

the primitive reduced cell. It is thus possible to

calculate a series of three-dimensional patterns and

Plans for shell instruments along a chosen axis, 1950-

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resolved to lead to a satisfactory response behavior.

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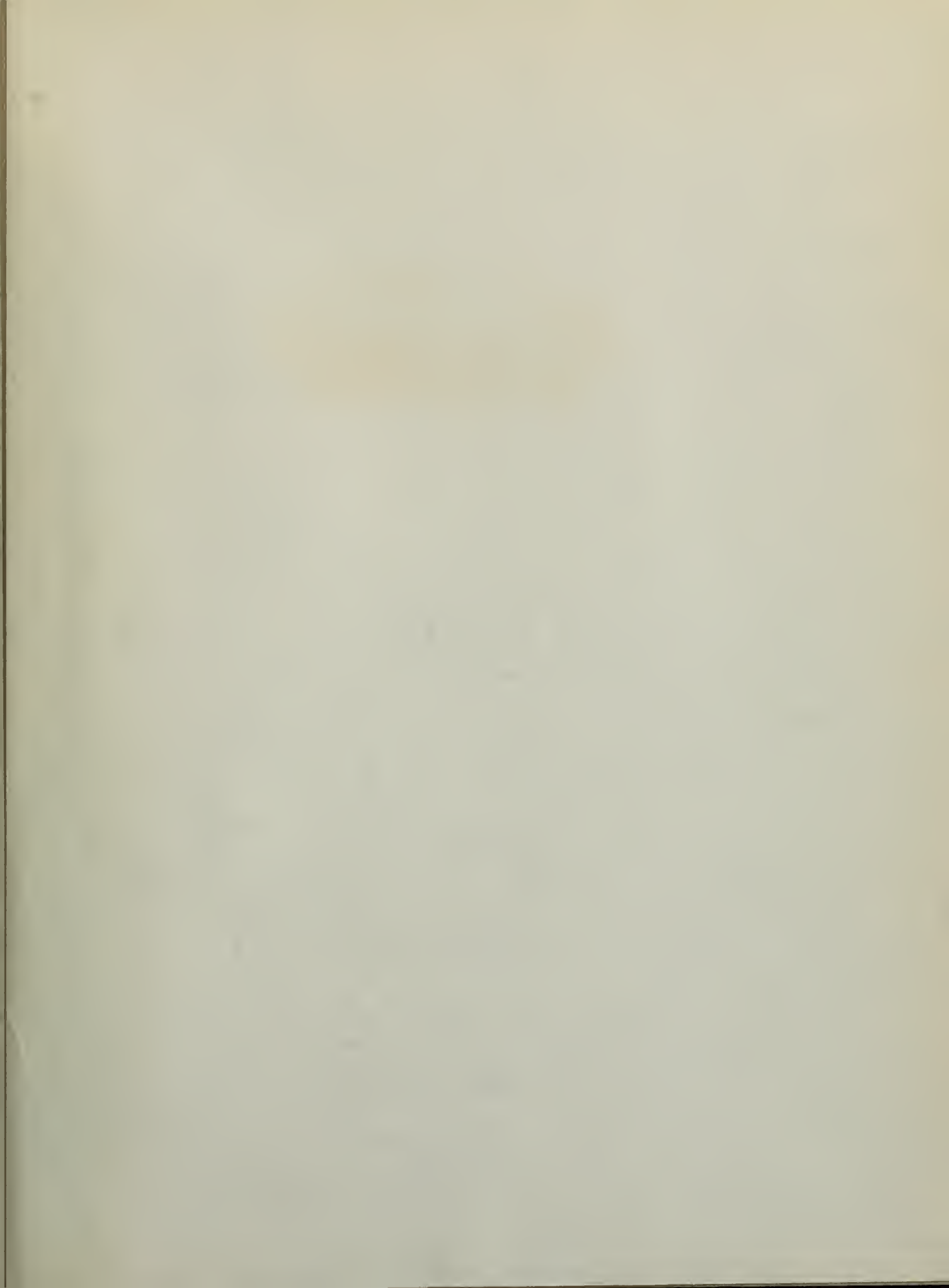
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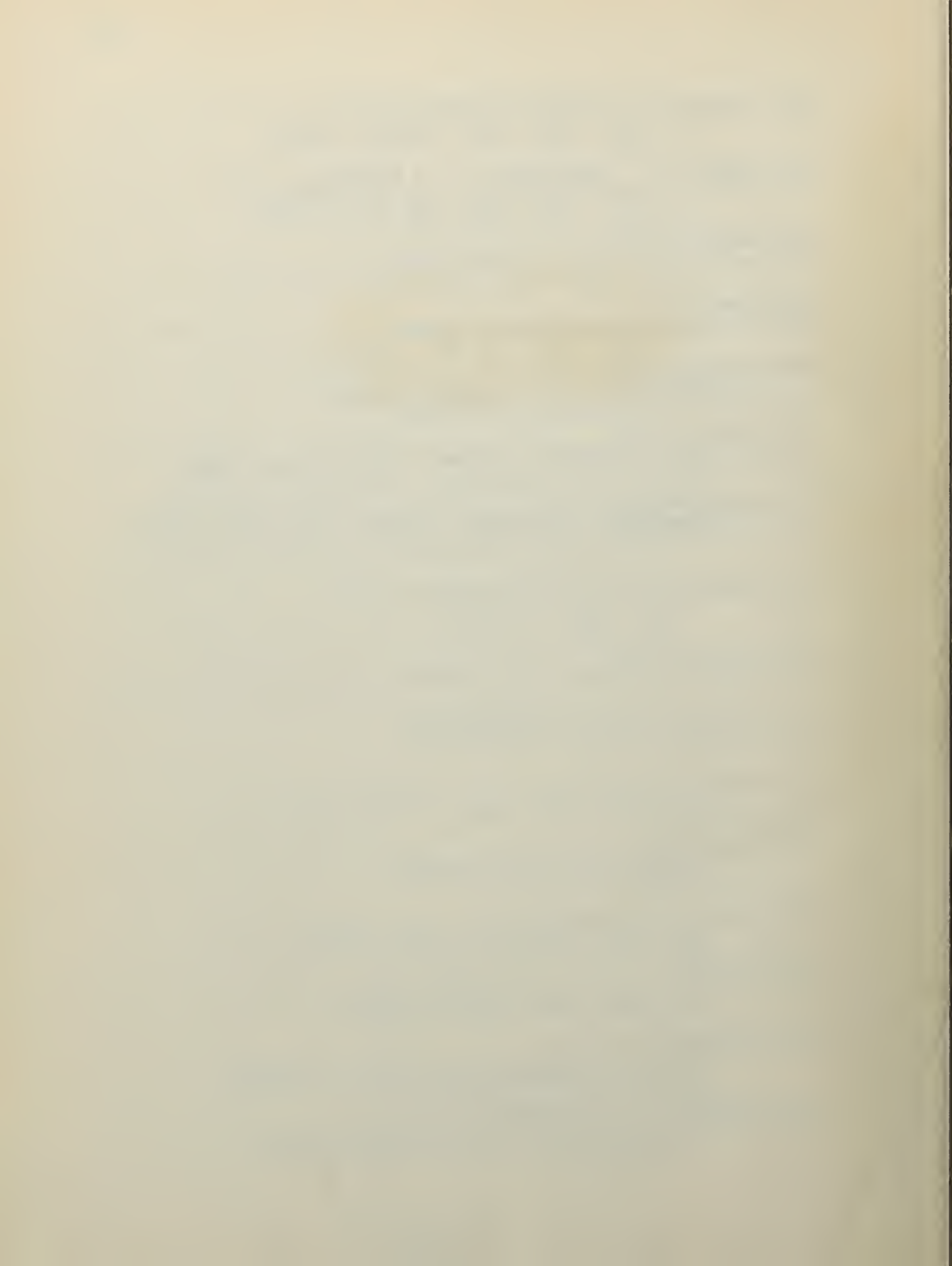
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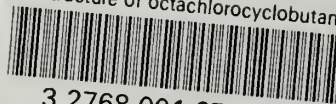
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